

U.S. Department of  
Homeland Security

United States  
Coast Guard



## **U.S. Coast Guard**

### **Quality Assurance Project Plan Addendum (Rev. 1.0)**

#### **- Groundwater and Soil**

**USCG Atwater Facility  
Detroit, Michigan**

April 2011




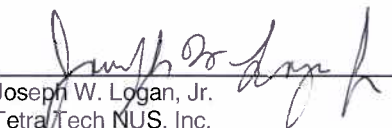


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**Quality Assurance Project Plan  
Addendum (Rev. 1.0)**

- Groundwater and Soil

Atwater Facility  
Detroit, Michigan

Prepared for:  
U.S. Coast Guard  
Civil Engineering Unit Cleveland

Prepared by:  
Tetra Tech NUS, Inc.

Project number:  
112G02435

Date:  
April 2011



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AOC	Administrative Order of Consent
ARAR	Applicable or relevant and appropriate requirement
ASTM	American Society for Testing and Materials
AVS	Acid volatile sulfides
AVS/SEM	Acid volatile sulfides/simultaneously extracted metals
BERA	Baseline Ecological Risk Assessment
°C	Degrees Celsius
CA	Corrective action
CAL	Calibration
CCV	Continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CF	Calibration factor
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COB	Close of business
COC	Chain-of-custody
CQA	Construction quality assurance
CSM	Conceptual Site Model
CSV	Comma-separated value



## Acronyms

DQI	Data quality indicator
DQO	Data quality objective
ECO RA	Ecological risk assessment
EDD	Electronic data deliverable
ERA	Ecological Risk Assessment
FAA	Flame Atomic Absorption
FSP	Field Sampling Plan
FSPG&S	Field Sampling Plan – Groundwater and Soil
GIS	Geographic Information System
GPS	Global Positioning System
HHRA	Human health risk assessment
HSP	Health and Safety Plan
ICV	Initial calibration verification
ID	Identification
IDW	Investigation-derived waste
L	Liter
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
MDEQ	Michigan Department of Environmental Quality
MDL	Method detection limit



MDNR	Michigan Department of Natural Resources
mg/kg	Milligram per kilogram
mg/L	Milligram per liter
MS	Matrix spike
MSD	Matrix spike duplicate
µg/L	Microgram per liter
NA	Not Applicable
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NEIC	National Enforcement Investigations Center
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
ppm	Part per million
PQO	Project quality objectives
QA	Quality assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QAPPA	Quality Assurance Project Plan Addendum
QA/QC	Quality assurance/quality control



## Acronyms

QC	Quality control
RAO	Removal action objective
RSE	Removal site evaluation
RL	Reporting limit
RPD	Relative percent difference
RPM	Remedial Project Manager
RRD	Remediation and Redevelopment Division
RSD	Relative standard deviation
SAP	Sampling and Analysis Plan
SDG	Sample delivery group
SOP	Standard Operating Procedure
SOW	Statement of Work
SRI	Supplemental Remedial Investigation
TBD	To be determined
TCP	Traffic Control Plan
TCLP	Toxicity Characteristic Leaching Procedure
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
%D	Percent difference



%R

Percent recovery



## **1. Introduction**

This Quality Assurance Project Plan Addendum (QAPPA) has been developed to meet the data quality requirements of the site evaluation and soil removal action at the U.S. Coast Guard (USCG) Atwater Facility (Site). It provides details on the sampling and analytical methods and procedures that will be used during site evaluation and soil removal action at the Site, if warranted. This QAPPA is intended to guide site characterization activities to be consistent with the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 Code of Federal Regulations (CFR) Part 300 and to supplement the site-specific field sampling plan (FSP) and/or removal action work plan (RAWP) for the USCG lighthouse divestiture program, and will be modified as necessary as the sampling program develops. The U.S. Environmental Protection Agency (USEPA) has concurred with the FSP and RAWP.

### **1.1 Development of the Quality Assurance Project Plan Addendum**

As described on the USEPA's Quality System Website (<http://www.epa.gov/QUALITY/qapps.html>), the purpose of a QAPP is to document the planning, implementation and assessment procedures for a particular project, as well as any specific quality assurance and quality control (QA/QC) activities. It is to integrate all the technical and quality aspects of the project to provide a "blueprint" for obtaining the type and quality of environmental data and information needed for a specific decision or use.

In March 2005, the Intergovernmental Data Quality Task Force developed a policy and guidance document to fulfill the project-specific requirements of Part B of the American National Standards Institute, American Society for Quality Control standard, Specifications and Guidelines for Environmental Data Collection and Environmental Technology Programs (ANSI/ASCQ E4) to ensure that federal departments and agencies will produce consistent QAPPs that reflect a systematic planning approach to the collection and use of environmental data. That guidance document—the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) Manual V1, March 2005 (USEPA 2005a)—was followed in the development of the March 2010 QAPP and this QAPPA.

### **1.2 Project Setting**

Activity at the USCG Atwater Facility property began prior to 1897 in Detroit, Michigan (see Figure 1 and Figure 2). The property space covers approximately 1.26 acres of harbor front land in downtown Detroit. No buildings are currently on the site. The former maintenance building and garage were demolished around 2004. The site is predominantly covered with asphalt with a small grassy area. A fence with a locked gate is located along Atwater Street



and a fence is located on the eastern and western sides of the property to restrict access to the site.

The subject property has been used as a USCG station since the late 1800s. Land use prior to USCG ownership is unknown. Dating back to the late 1800s, the historic site structures including a boathouse, two slips, a boat shop, various garages, and office type buildings were present on site.

Currently the property is vacant and no structures remain onsite.

Two environmental site assessments were conducted at the Atwater site. The first was a Phase I and II Environmental Site Assessment for Detroit Atwater conducted by Tetra Tech NUS, Inc. December 2002 (Table 1). This assessment identified four potential areas of concern: possible asbestos - containing building materials, the AST, oil and gas storage area, and the filled - in slip area. TtNUS then conducted a Phase II investigation where seven soil samples were collected. Samples were analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and RCRA metals. Although a few SVOCs and metals exceeded MDEQ Michigan residential soil criteria results were within an acceptable range. Samples were collected in various locations laterally around the property and up to 9 feet below ground surface (bgs). At the time of those investigations, both the maintenance building and garage structures were present on site. Based on the sample results there were no areas of significant environmental concern requiring rectification prior to transfer of the property and had no further recommendations at that time (TtNUS, 2002)

The second a Phase II Environmental Site Assessment prepared for the Economical Development Corporation of the City of Detroit, was conducted by Enviro Matrix in July 2006. The Phase I indicated three potential RECs exist at the site. The RECs identified were: fill of unknown origin may have been brought into the parcel, the property has been used by the USCG and other unknown uses dating back to the late 1800s, and a history of heavy industrial activities in the area. Subsequently, they conducted a Phase II site investigation five soil borings were advanced and six soil samples were collected for polycyclic aromatic hydrocarbons (PAHs), metals (Michigan 10) and one location was analyzed for VOCs. This investigation identified elevated PAHs and metals greater than the most restrictive MDEQ. Samples were collected in various locations laterally around the property and up to 10.5 feet bgs (Enviro Matrix, 2006). At the time of this investigation, both the maintenance building and garage structures had been demolished and building footprints were included in the investigation.



During the April 2010 investigation by TtNUS, fifteen borings were advanced and soil samples were collected at two depth intervals: immediately below the surface (approximately 0 to 2 feet bgs) and immediately above the water table (approximately 3 to 5 feet bgs). These samples were analyzed for metals and PAHs, and several contaminants were detected at concentrations greater than Michigan DNRE criteria (Figure 4). Based on this information and other field observations, USEPA requested the installation of permanent monitoring wells.

### **1.3 Applicability of the Quality Assurance Project Plan Addendum**

This QAPPA is intended primarily for use in collection of environmental data for the following purpose:

- To characterize the groundwater at the site.

Specific requirements for the soil and groundwater investigation and removal activities at the Site are described in the FSPG&S, TtNUS, 2011.

### **1.4 Site Evaluation Activities**

Details of the Site evaluation sample program are provided in the FSPG&S, TtNUS, 2011.

PAHs and metal impacts to soil are present based on previous investigations and historic site activities. Groundwater at the site will be characterized.

### **1.5 Removal Action Activities**

At this time removal action activities are not anticipated.

## **2. Document Overview**

The UFP-QAPP guidance (USEPA 2005a) includes a series of 37 worksheets that can be used to present the critical information required in a QAPP. Because the worksheets were designed to “ensure consistent content and presentation of information in a project-specific QAPP... and streamline the review of QAPPs by regulators and others” (USEPA 2005a), this QAPPA has been developed using these worksheets, which provide information associated with four key topics:



- Project Management and Objectives
- Measurement and Data Acquisition
- Assessment and Oversight
- Data Review

A series of attachments is also included with this QAPPA. These attachments include the following information:

- Chain-of-custody forms.
- Laboratory certifications.
- Laboratory Quality Assurance Manual.
- Standard Operating Procedures (SOPs).

Only the worksheets that are necessary for the groundwater sampling tasks are included in this QAPPA. Worksheets in the March 2010 QAPP that are also applicable to groundwater sampling tasks but do not require changes are not included in the QAPPA. Note that for clarity, some QAPPA worksheets refer to the re-sampling of several boring locations (see FSPG&S, TtNUS, 2011).

When elements required by the UFP-QAPP are present in other documents (e.g., SOPs), careful cross-referencing of these other documents can be used in lieu of repeating information (USEPA 2005a). Following this guidance, this QAPPA provides certain relevant information by referencing the FSPG&S, TtNUS, 2011. All of these planning documents are companion documents to the QAPPA.



### **3. References**

NOAA, 2011. *Stream Gauge Data*. <http://www.great-lakes.net/envt/water/levels/levels-cur/stclairwlc.html>

TtNUS, 2010. *Quality Assurance Project Plan*. March 2010.

TtNUS, 2011. *Field Sampling Plan for Site Evaluation – Groundwater and Soil, USCG Atwater Facility*. April 2011.

United States Environmental Protection Agency. 2005a. Intergovernmental Data Quality Task Force. *Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. Part 1: UFP-QAPP Manual*. Version 1, March 2005. Publication Numbers: USEPA: EPA-505-B-04-900A, Department of Defense: DTIC ADA 427785. [http://www.epa.gov/fedfac/pdf/ufp\\_qapp\\_v1\\_0305.pdf](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf).

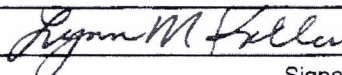
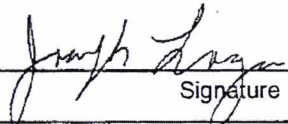
United States Environmental Protection Agency. 2005b. Intergovernmental Data Quality Task Force. *Uniform Federal Policy for Quality Assurance Project Plans. Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities*. Version 1, March 2005. Publication Numbers: USEPA: EPA-505-B-04-900B, Department of Defense: DTIC ADA 426957. [http://www.epa.gov/swerffrr/pdf/qaqc\\_v1\\_0305.pdf](http://www.epa.gov/swerffrr/pdf/qaqc_v1_0305.pdf).



## Worksheets

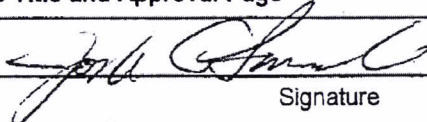
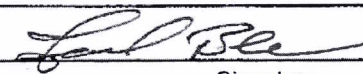


## QAPP Worksheet #1G Title and Approval Page

Site Name/Project Name:	Atwater Facility
Site Location:	Harborfront land property in downtown Detroit, Wayne County, Michigan. Companion document Field Sampling Plan Groundwater and Soil (FSPG&S) Tetra Tech NUS, Inc., 2011; Figures 1 and 2 provide the site locations.
Document Title:	U.S. Coast Guard Quality Assurance Project Plan Addendum – Groundwater and Soil, USCG Atwater Facility, Detroit, Michigan
Lead Organization:	U.S. Coast Guard
Preparer's Name and Organizational Affiliation	Joseph Logan, Tetra Tech NUS, Inc. / Shannon Hill, Tetra Tech NUS, Inc.
Preparer's Contact Information:	661 Andersen Drive, Pittsburgh, PA 15220 412-921-7090; <a href="mailto:Joe.Logan@tetrattech.com">Joe.Logan@tetrattech.com</a> / <a href="mailto:Shannon.Hill@tetrattech.com">Shannon.Hill@tetrattech.com</a>
Preparation Date:	December 2009 to April 2011
Lead Organization's Project Manager	 Signature Lynn Keller, USCG Project Manager
Investigative Organization's Project Coordinator	 Signature Joseph Logan, Tetra Tech NUS, Inc.



QAPP Worksheet #1G Title and Approval Page

Investigative Organization's Project Quality Assurance Manager:	
	Signature
	Joseph Samchuck, Tetra Tech NUS, Inc.
Lead Organization's Program Manager:	
	Signature
	Frank Blaha, USCG Program Manager

Document Control Number:



**QAPP Worksheet #2G QAPP Identifying Information**

<b>Site Name/Project Name:</b>	Atwater Facility
<b>Site Location:</b>	2660 East Atwater Street, Detroit, Wayne County, Michigan.
<b>Site Number/Code:</b>	NA
<b>Operable Unit:</b>	NA
<b>Contractor Name:</b>	Tetra Tech NUS, Inc.
<b>Contractor Number:</b>	HSCG83-08-D-3CL109
<b>Contract Title:</b>	CERCLA Investigation
<b>Work Assignment Number:</b>	HSCG83-09-J-3CL358
<b>Identify guidance used to prepare QAPP:</b>	Uniform Federal Policy for Quality Assurance Project Plans, Manual VI (2005)
<b>Identify regulatory program:</b>	Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)
<b>Identify approval entity:</b>	NA
<b>Indicate whether the QAPP is a generic or a project-specific QAPP?</b>	This QAPPA is a project-specific QAPP intended to serve the needs of the Site Evaluation and Removal Action activities, as described in the FSPG&S, Tetra Tech NUS, Inc., 2011.
<b>List dates of scoping sessions that were held:</b>	June 20, 2007 at USEPA Region 5 offices in Chicago, Illinois Understanding of Scope of Services dated September 2009



**QAPP Worksheet #2G QAPP Identifying Information**

<b>List dates and titles of QAPP documents written for previous site work, if applicable:</b>	
<b>Quality Assurance Project Plan</b>	QAPP, March 2010
<b>List organizational partners (stakeholders) and connection with lead organization:</b>	U.S. Coast Guard – Lead Agency
<b>List data users:</b>	U.S. Coast Guard Project Team
<b>Lead Organization's Program Manager:</b>	Frank Blaha, U.S. Coast Guard, Program Manager



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
<b>Project Management and Objectives</b>		
2.1 Title and Approval Page	- Title and Approval Page	- Worksheet #1G Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents  - QAPP Identifying Information	- The Table of Contents is provided following the QAPP cover page.  - Worksheet #2G QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	- Worksheet #3G Distribution List/ Personnel Sign-Off  Note: Worksheet 3G and 4G combined



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	<ul style="list-style-type: none"> <li>- Project Organizational Chart</li> <li>- Communication Pathways</li> <li>- Personnel Responsibilities and Qualifications Table</li> <li>- Special Personnel Training Requirements Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #5G Project Organization Chart</li> <li>- Worksheet #6 Communication Pathways</li> <li>- Worksheet #7 Personnel Responsibilities and Qualifications and</li> <li>- Worksheet #8G Special Personnel Training Requirements</li> </ul>
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History and Background	<ul style="list-style-type: none"> <li>- Project Planning Session Documentation (including Data Needs tables)</li> <li>- Project Scoping Session Participants Sheet</li> <li>- Problem Definition, Site History and Background</li> <li>- Site Maps (historical and present)</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #9G Project Team Planning Sessions Participants' Sheet and</li> <li>- Worksheet #10G Problem Definition for Project Data Quality Objectives)</li> </ul> <p>Site history and more detail concerning the project Data Quality Objectives can be found in the FSPG&amp;S, Tetra Tech NUS, Inc., 2011.</p> <p>Site maps are provided in the FSPG&amp;S, Tetra Tech NUS, Inc., 2011. Prepared for the Site.</p>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	<ul style="list-style-type: none"> <li>- Site-Specific Project Quality Objectives</li> <li>- Measurement Performance Criteria Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #11G Project Quality Objectives/Systematic Planning Process Statements and</li> <li>- Worksheet #12-1G Measurement Performance Criteria for project analytes</li> </ul>
2.7 Secondary Data Evaluation	<ul style="list-style-type: none"> <li>- Sources of Secondary Data and Information</li> <li>- Secondary Data Criteria and Limitations Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #13 Secondary Data Criteria and Limitations</li> </ul>
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	<ul style="list-style-type: none"> <li>- Summary of Project Tasks</li> <li>- Reference Limits and Evaluation Table</li> <li>- Project Schedule/Timeline Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #14G Summary of Project Tasks</li> <li>- Worksheet #15-2G Reference Limits and Evaluation for specific monitoring activities and</li> <li>- Worksheet #16G Project Schedule/Timeline</li> </ul>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
<b>Measurement/Data Acquisition</b>		
3.1 Sampling Tasks		
3.1.1 Sampling Process Design and Rationale	- Sampling Design and Rationale	- Worksheet #17G Sampling Design and Rationale
3.1.2 Sampling Procedures and Requirements	- Sample Location Map	- Worksheet #18G Sampling Locations and Methods/Standard Operating Procedure Requirements for the project
3.1.2.1 Sampling Collection Procedures	- Sampling Locations and Methods/Standard Operating Procedure Requirements Table	- Worksheet #18G Sampling Locations and Methods/Standard Operating Procedure Requirements for the project
3.1.2.2 Sample Containers, Volume and Preservation	- Analytical Methods/Standard Operating Procedure Requirements Table	- Worksheet #19G Analytical Standard Operating Procedure Requirements (Sample Containers Preservation and Holding Times)
3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures	- Field Quality Control Sample Summary Table	- Worksheet #20G Sample Quantities and Control Frequencies
3.1.2.4 Field Equipment Calibration, Maintenance, Testing and Inspection Procedures	- Sampling Standard Operating Procedures	- Worksheet #21G Field Sampling Standard Operating Procedure References
3.1.2.5 Supply Inspection and Acceptance Procedures	- Field Equipment Calibration, Maintenance, Testing and Inspection Table	- Worksheet #22G Field Equipment Calibration, Maintenance, Testing, and Inspection
3.1.2.6 Field Documentation Procedures		More details concerning the sampling design, rational and procedures can be found in the FSPG&S, Tetra Tech NUS, Inc., 2011.



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	<ul style="list-style-type: none"> <li>- Analytical Standard Operating Procedures</li> <li>- Analytical Standard Operating Procedure References Table</li> <li>- Analytical Instrument Calibration Table</li> <li>- Analytical Instrument and Equipment Maintenance, Testing and Inspection Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #23G Analytical Standard Operating Procedure References</li> <li>- Worksheet #24 Analytical Instrument Calibration</li> <li>- Worksheet #25 Analytical Instrument and Equipment Maintenance, Testing and Inspection</li> </ul> <p>The analytical Standard Operating Procedures can be found in Attachment T-1</p>
3.3 Sample Collection Documentation, Handling, Tracking and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	<ul style="list-style-type: none"> <li>- Sample Collection Documentation Handling, Tracking and Custody Standard Operating Procedures</li> <li>- Sample Container Identification</li> <li>- Sample Handling Flow Diagram</li> <li>- Example Chain-of-Custody Form and Seal</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #26 Sample Handling System</li> <li>- Worksheet #27G Sample Custody Requirements</li> <li>- An example of the Chain-of-Custody form can be found in Attachment 1</li> </ul> <p>More details concerning the field sampling procedures can be found in the FSPG&amp;S, Tetra Tech NUS, Inc., 2011.</p>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	<ul style="list-style-type: none"> <li>- QC Samples Table</li> <li>- Screening/Confirmatory Analysis Decision Tree</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #28-1G presents Quality Control sample information for project analytes</li> </ul>
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	<ul style="list-style-type: none"> <li>- Project Documents and Records Table</li> <li>- Analytical Services Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #29G Project Documents and Records</li> <li>- Worksheet #30G Analytical Services</li> </ul>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
<b>Assessment/Oversight</b>		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	<ul style="list-style-type: none"> <li>- Assessments and Response Actions</li> <li>- Planned Project Assessments Table</li> <li>- Audit Checklists</li> <li>- Assessment Findings and Corrective Action Responses Table</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #31 Planned Project Assessments</li> <li>- Worksheet #32 Assessment Findings and Corrective Action Responses</li> </ul> <p>Laboratory Certifications can be found in Attachment 2</p>
4.2 QA Management Reports	<ul style="list-style-type: none"> <li>- Conformance of project activities to QAPP requirements and procedures</li> <li>- Status of project and schedule delays</li> <li>- Deviations from the approved QAPP and approved amendments to the QAPP</li> <li>- Results of data review activities</li> <li>- Required corrective actions and effectiveness of corrective action implementation</li> <li>- Data usability assessments in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #33 Quality Assurance Management Reports</li> </ul>
4.3 Final Project Report	<ul style="list-style-type: none"> <li>- All Quality Assurance Management Reports attached</li> <li>- Additional data quality concerns and resolution documentation</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #33 Quality Assurance Management Reports</li> </ul>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	<ul style="list-style-type: none"> <li>- Verification (Step I) Process Table</li> <li>- Validation (Steps IIa and IIb) Process Table</li> <li>- Validation (Steps IIa and IIb) Summary Table</li> <li>- Usability Assessment</li> </ul>	<ul style="list-style-type: none"> <li>- Worksheet #34 Verification (Step I) Process</li> <li>- Worksheet #35 Validation (Steps IIa and IIb) Process</li> <li>- Worksheet #36G Validation (Steps IIa and IIb) Summary</li> <li>- Worksheet #37 Usability Assessment</li> </ul>



**QAPP Worksheet #2G QAPP Identifying Information**

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining	None	NA

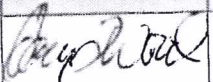
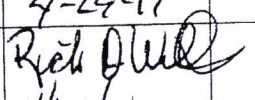


## QAPP Worksheet #3G Distribution List/Project Personnel Sign-Off

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	Email Address	Signature/Date QAPP Read	Document Control Number
Frank Blaha	Program Manager	USCG	216.902.6255	216.902.6277	<a href="mailto:Frank.A.Blaha@uscg.mil">Frank.A.Blaha@uscg.mil</a>	<i>Frank A. Blaha</i> 4/27/11	NA
Lynn Keller	Project Manager	USCG	216.902.6258	216.902.6277	<a href="mailto:Lynn.M.Keller@uscg.mil">Lynn.M.Keller@uscg.mil</a>	<i>Lynn M. Keller</i> 4/26/11	NA
Owen Thompson	USEPA Remedial Project Manager	USEPA Region 5	312.886.4843	312.353.8426	<a href="mailto:Thompson.Owen@epa.gov">Thompson.Owen@epa.gov</a>	<i>Owen Thompson</i> 4/27/11	NA
Joseph Logan	Project Coordinator	Tetra Tech NUS, Inc.	412.921.7231	412.921.4040	<a href="mailto:Joe.Logan@tetrattech.com">Joe.Logan@tetrattech.com</a>	<i>Joseph Logan</i> 4/26/11	NA
Joseph Samchuck	Data Quality Assurance Manager	Tetra Tech NUS, Inc.	412.921.8510	412.921.4040	<a href="mailto:Joseph.Samchuck@tetrattech.com">Joseph.Samchuck@tetrattech.com</a>	<i>Joseph Samchuck</i> 4/26/11	NA



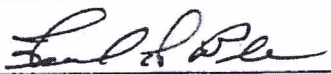
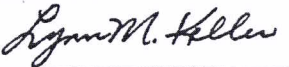
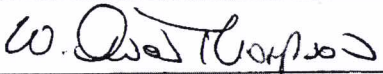
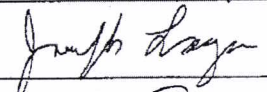

**QAPP Worksheet #3G Distribution List/Project Personnel Sign-Off**

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Signature/Date QAPP Read	Document Control Number
Gary Wood	Laboratory Project Manager	TriMatrix Laboratories, Inc.	616.940.4206	616.942.7463	<a href="mailto:WoodGL@trimatrixlabs.com">WoodGL@trimatrixlabs.com</a>	 4-29-11	NA
Rick Wilburn	Laboratory Quality Assurance Manager	TriMatrix Laboratories, Inc.	616.940.4246	616.942.7463	<a href="mailto:WilburnR@trimatrixlabs.com">WilburnR@trimatrixlabs.com</a>	 4/29/11	NA

**Note:** Copies of the QAPPA will be distributed to the individuals above. The copies will consist of the following documents: QAPPA and any subsequent QAPPA revisions and addendums. The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

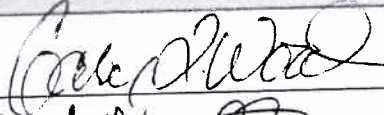
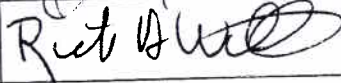


**QAPP Worksheet #4G Project Personnel Sign-Off**

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Frank Blaha	Program Manager	216.902.6255		4/27/11
Lynn Keller	Project Manager	216.902.6258		4/26/11
Owen Thompson	USEPA Remedial Project Manager	312.886.4843		4/27/11
Joseph Logan	Project Coordinator	412.921.7231		4/26/11
Joseph Samchuck	Data Quality Assurance Manager	412.921.8510		4/26/11

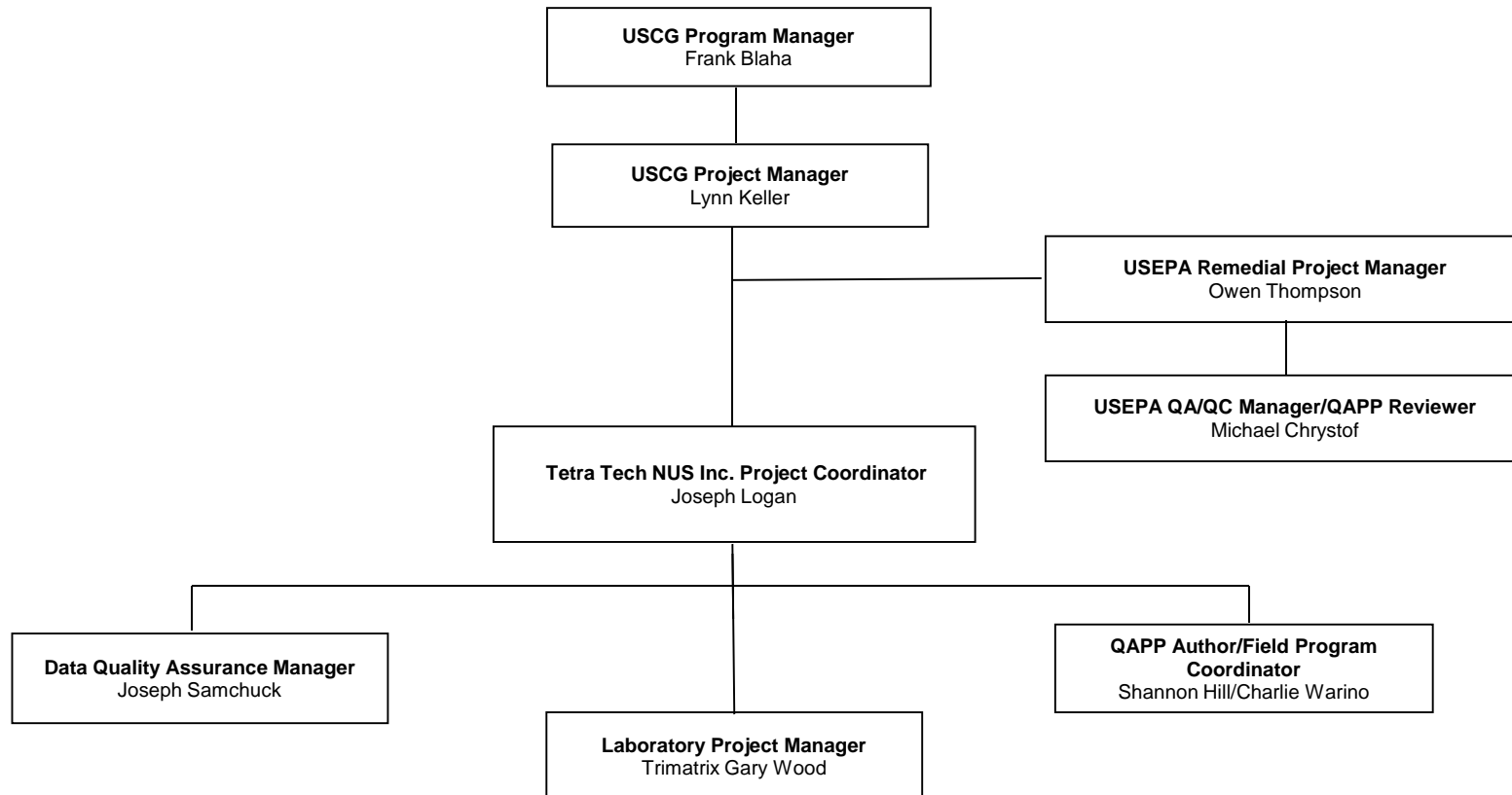


QAPP Worksheet #4G Project Personnel Sign-Off

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Gary Wood	Laboratory Project Manager	616.940.4206		4-29-11
Rick Wilburn	Laboratory Quality Assurance Manager	616.940.4246		4/29/11



**QAPP Worksheet #5G Project Organization Chart**





**QAPP Worksheet #8G Special Personnel Training Requirements**

<b>Project Function</b>	<b>Specialized Training</b>	<b>Training Provider</b>	<b>Training Date</b>	<b>Personnel/Groups Receiving Training</b>	<b>Personnel Titles/ Organizational Affiliation</b>	<b>Location of Training Records/Certificates</b>
Field Activities	40-hour HAZWOPER and Associated 8-hour Refresher	Certified Trained Professionals	Not Applicable	Field operations personnel	Consultant Field Staff and Subcontractors (Driller)	Tetra Tech NUS, Inc. project offices and Site-Specific Health and Safety Plan
Analytical Chemistry	State Certification of Laboratory	Michigan	Not Applicable	Not Applicable	TriMatrix Laboratories Inc., 5560 Corporate Exchange Court SE, Grand Rapids, MI 49512 616.975.4500	TriMatrix Laboratories Inc., 5560 Corporate Exchange Court SE, Grand Rapids, MI 49512 616.975.4500
Additional training/certification requirements are listed in the project health and safety plan Tetra Tech NUS, Inc., 2010.						

**Note:** Current HAZWOPER training certificates for consultant field staff and subcontractors will be maintained in the Tetra Tech NUS, Inc., Pittsburgh PA office location for each employee performing work at the Site where 40-hour training is required for the position assignment.



**QAPP Worksheet #9G Project Team Planning Sessions Participants' Sheet**

Project Name: <b>USCG (Generic QAPP) Project planning session and Site Specific Kick-off meeting</b>			Site Name: <b>Atwater Facility</b>		
Projected Date(s) of Sampling: May 2011			Site Location:		
Project Manager: Lynn Keller			Detroit, Michigan		
Date of Session: June 20, 2007; Kick-off meeting: September 28, 2009 and August 16, 2010					
Scoping Session Purpose: Review QAPP preparation requirements and approach associated with the creation of a QAPP applicable to USGC lead in soil sites for site evaluation and removal action.					
Name	Title	Affiliation	Phone	E-mail Address	Project Role
Michael Chrystof	Remedial Project Quality Assurance/Quality Control Manager	USEPA	312.353.3705	<a href="mailto:Chrystof.Michael@epa.gov">Chrystof.Michael@epa.gov</a>	QAPP Reviewer
Frank Blaha	Chief, Environmental Compliance	USCG CEU Cleveland	216.902.6255	<a href="mailto:Frank.A.Blaha@uscg.mil">Frank.A.Blaha@uscg.mil</a>	Program Manager
Lynn Keller	USCG Project Manager	USCG	216.902.6258	<a href="mailto:Lynn.M.Keller@uscg.mil">Lynn.M.Keller@uscg.mil</a>	Project Manager
Joseph Logan	Project Coordinator	Tetra Tech NUS, Inc.	412.921.7231	<a href="mailto:Joe.Logan@tetrattech.com">Joe.Logan@tetrattech.com</a>	Project Coordinator
Meeting Discussions/Comments: Meeting Discussions/Comments: The scoping session and kick-off meeting were conducted by conference call. The call discussed the SOW provided by the USCG and implementation of the SOW.					



### QAPP Worksheet #10G Problem Definition — Data Quality Objectives

#### Step 1: State the Problem:

The problem to be addressed is the presence of PAH and metals impacts in the groundwater from possible historical USCG on-site activities. Previous investigations at the Site determined that surface and subsurface soil PAH and metal contamination is present. This investigation will provide further information about the onsite contamination. (See FSPG&S, Tetra Tech NUS, Inc., 2011).

#### Step 2: Identify the Goal of the Study:

- Generate information sufficient to complete the evaluation of the Site to determine the nature and extent of the PAH and metals impacts in groundwater related to historic site use.

#### Step 3: Identify Information Inputs:

The following new data will be collected to further define the potential groundwater impacts resulting from lead impacts at the Site.

- Collection of groundwater samples.



**Step 4: Define the Boundaries of the Sampling:**

Harbor front land property in downtown Detroit, Michigan.

**Step 5: Develop the Analytic Approach:**

The decision on whether new and historical data can be used in the exposure evaluation will be based on the validation results of the new sampling data. Following validation, the data will be flagged, as appropriate, and any use restrictions noted. The sampling plan has been devised so that the loss of any single data point will not hinder description of the distribution of constituents of concern. Given this, a reasonable decision rule would be that 90% of the data points not be rejected and deemed unusable for exposure evaluation purposes. The usable data will be evaluated versus the performance documented in Worksheet #15-2G. The required reporting limits are also documented in these tables so that the lowest achievable detection limit will be reported by the laboratory and, when possible, at or below the guidance values and cleanup target levels. Applicable actions will be evaluated, if needed, based on the results of the exposure evaluation.

**Step 6: Specify Performance or Acceptance Criteria:**

Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability and 2) understanding the representative nature of the sampling design. The sampling and analysis program described in the FSPG&S, Tetra Tech NUS, Inc., 2011 was the basis for preparation of this QAPPA in regards to investigation sampling requirements. Corrective actions are described within this document. The representative nature of the sampling design has been assured by discussions among professionals familiar with the Site and the appropriate government agencies.

**Step 7: Develop the Plan for Obtaining Data:**

The overall quality assurance objective is to develop and implement procedures for field sampling — chain of custody, laboratory analysis and reporting — that will provide results to support the evaluation of site data consistent with National Contingency Plan (NCP) requirements. Specific procedures for sampling, chain of custody, laboratory instrument calibration, laboratory analysis, data reporting, internal quality control, audits, preventive maintenance of field equipment and corrective action are described in other sections of this QAPPA.

The FSPG&S, Tetra Tech NUS, Inc., 2011 involves a phased approach to both sampling and analysis, including field screening and collection of samples for laboratory analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the understanding of PAH and metal distributions at the Site afforded by the level of sampling to be conducted.



### QAPP Worksheet #11G Project Quality Objectives/Systematic Planning Process Statements

<b>Who will use the data?</b>
Project team, their subcontractors, and overseeing agencies will use the data to assess the efficacy of the Site evaluation and removal action if warranted, and to confirm adherence to the performance standards.
<b>What will the data be used for?</b>
The data will be used to characterize PAH and metals concentrations in groundwater and their impacts, and if impacts are present above the applicable land use criteria, which removal action would potentially be performed.
<b>What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)</b>
<p>Data include Site evaluation samples of the Site groundwater.</p> <p>Standard protocols for sample collection and handling, sample preparation, and analytical methods will be followed. Standard operating procedures are provided in this document and in the FSPG&amp; S, Tetra Tech NUS, Inc., 2011</p>
<b>How “good” do the data need to be in order to support the environmental decision?</b>
Site characterization data need to complete the evaluation of the nature and extent of soil PAH and metal impacts at the Site, and determine if removal action will be necessary.



**QAPP Worksheet #11G Project Quality Objectives/Systematic Planning Process Statements**

<b>How much data are needed? (number of samples for each analytical group, matrix and concentration)</b>
The number of samples and analyses for each media are summarized in Worksheet #20 and described in the FSPG&S, Tetra Tech NUS, Inc., 2011.
<b>Where, when, and how should the data be collected/generated?</b>
<p>Groundwater samples will be collected using low-flow purging and sampling techniques with either a peristaltic pump or decontaminated submersible pump. Groundwater data will be generated by laboratory analysis of PAH and metals using (filtered and unfiltered) USEPA Method 8270C and SW-846 6020A and 7470A (mercury).</p> <p>The specific sampling program is described in FSPG&amp;S, Tetra Tech NUS, Inc., 2011. Standard protocols for sample collection and handling, sample preparation, and analytical methods will be followed. Standard operating procedures for field sampling are referenced in this document and provided in the FSPG&amp;S, Tetra Tech NUS, Inc., 2011.</p>
<b>Who will collect and generate the data?</b>
Project team.
<b>How will the data be reported?</b>
Site evaluation data will be reported in an Engineering Evaluation/Cost Analysis for the Site.
<b>How will the data be archived?</b>
All data will be archived by USCG CEU Cleveland in their Cleveland, Ohio office.



**QAPP Worksheet #12-1G Measurement Performance Criteria – Metals (Groundwater)**

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	Metals				
<b>Concentration Level</b>	All				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/Standard Operating Procedures<sup>2</sup></b>	<b>Data Quality Indicators</b>	<b>Measurement Performance Criteria</b>	<b>Quality Control Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
FSPG&S, Tetra Tech NUS, Inc., 2011	6020A/ GR-01-129	Precision — Overall	RPD < 30%	Field duplicate	S&A
		Accuracy/Bias Contamination	Analytes < ½ RL	Blanks (field, equipment, calibration, preparation)	S&A
		Accuracy/Bias	%R (90-110)	Initial calibration verification	A
		Accuracy/Bias	%Difference ≤ 10%	Continuing calibration verification	A
		Precision — lab	ICS A: Less than absolute value of the LOD ICS AB: 80-120%	Interference check sample (A and AB)	A
		Accuracy/Bias	%R (80-120)	MS/MSD	A
		Precision	%RPD <20	MS/MSD	A
		Accuracy/Bias	%R (80-120)	LCS	A



**QAPP Worksheet #12-1G Measurement Performance Criteria – Metals (Groundwater)**

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	Metals				
<b>Concentration Level</b>	All				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/Standard Operating Procedures<sup>2</sup></b>	<b>Data Quality Indicators</b>	<b>Measurement Performance Criteria</b>	<b>Quality Control Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
		Accuracy/Bias	%R (75-125)	Post-digestion spike	A
		Precision	%D < 10%	Serial dilution <sup>3</sup>	A

**Notes:**

LCS Laboratory control sample.

MS Matrix spike.

MSD Matrix spike duplicate.

RL Reporting limit.

RPD Relative percent difference.

SOP Standard operating procedure.

%D Percent difference.

%R Percent recovery.

1. Reference number from QAPP Worksheet #21.

2. Reference number from QAPP Worksheet #23.

3. Performed as needed only for analytes with concentration > 100 times the method detection limit.



## QAPP Worksheet #12-1G Measurement Performance Criteria – Mercury (Groundwater)

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	Mercury				
<b>Concentration Level</b>	All				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/Standard Operating Procedures<sup>2</sup></b>	<b>Data Quality Indicators</b>	<b>Measurement Performance Criteria</b>	<b>Quality Control Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
FSPG&S, Tetra Tech NUS, Inc., 2011	7470A/ GR-01-123	Precision — Overall	RPD < 30%	Field duplicate	S&A
		Accuracy/Bias Contamination	Analytes < ½ RL	Blanks (field, equipment, calibration, preparation.)	S&A
		Accuracy/Bias	%R (80-120)	Initial calibration verification	A
		Accuracy/Bias	%Difference ≤ 20%	Continuing calibration verification	A
		Accuracy/Bias	%R (80-120)	MS/MSD	A
		Precision	%RPD <20	MS/MSD	A
		Accuracy/Bias	%R (80-120)	LCS	A

**Notes:**

LCS Laboratory control sample.  
MS Matrix spike.  
MSD Matrix spike duplicate.



RL Reporting limit.  
RPD Relative percent difference.  
SOP Standard operating procedure.  
%D Percent difference.  
%R Percent recovery.

1. Reference number from QAPP Worksheet #21.
2. Reference number from QAPP Worksheet #23.



## QAPP Worksheet #12-1G Measurement Performance Criteria – PAHs (Groundwater)

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	PAHs				
<b>Concentration Level</b>	All				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/Standard Operating Procedures<sup>2</sup></b>	<b>Data Quality Indicators</b>	<b>Measurement Performance Criteria</b>	<b>Quality Control Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
FSPG&S, Tetra Tech NUS, Inc., 2011	SW-846 8270C/GR-04-103	Precision — Overall	RPD < 30%	Field duplicate	S&A
		Accuracy/Bias Contamination	Analytes < ½ RL	Blanks (field, equipment, calibration, prep.)	S&A
		Accuracy/Bias	%R (80-120)	Initial calibration verification	A
		Accuracy/Bias	%D ± 20%	Continuing calibration verification	A
		Accuracy/Bias	%R within DoD QSM v4.1 control limits <sup>3</sup>	MS/MSD	A
		Precision	%RPD <30	MS/MSD	A
		Accuracy/Bias	%R within DoD QSM v4.1 control limits <sup>3</sup>	LCS	A
		Accuracy/Bias	See Worksheet 28-1G	Surrogates	A



**QAPP Worksheet #12-1G Measurement Performance Criteria – PAHs (Groundwater)**

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	PAHs				
<b>Concentration Level</b>	All				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/Standard Operating Procedures<sup>2</sup></b>	<b>Data Quality Indicators</b>	<b>Measurement Performance Criteria</b>	<b>Quality Control Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
		Accuracy/Bias	Retention time +/- 30 seconds from RT of the midpoint calibration initial calibration standard, and the area within -50% to +100 % of initial calibration midpoint standard.	Internal Standards	A

**Notes:**

LCS Laboratory control sample.

MS Matrix spike.

MSD Matrix spike duplicate.

RL Reporting limit.

RPD Relative percent difference.

SOP Standard operating procedure.

%D Percent difference.

%R Percent recovery.

PAH Polynuclear Aromatic Hydrocarbons

ICAL Initial Calibration

QSM DoD Quality Systems Manual, Version 4.1

1. Reference number from QAPP Worksheet #21.

2. Reference number from QAPP Worksheet #23G.

3. See Table G-6 QSM Version 4.1 (see Appendix A)



## Appendix A - 8270C PAH LCS/MS/MSD Control Limits

Analysis	Matrix	Analyte	Sur	Dup	MS	MSRPD	BS	BSRPD
8270C DoD PAHs	Water	Acenaphthene		30	45-110	30	45-110	30
8270C DoD PAHs	Water	Acenaphthylene		30	50-105	30	50-105	30
8270C DoD PAHs	Water	Anthracene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(a)anthracene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(a)pyrene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(b)fluoranthene		30	45-120	30	45-120	30
8270C DoD PAHs	Water	Benzo(k)fluoranthene		30	45-125	30	45-125	30
8270C DoD PAHs	Water	Benzo(g,h,i)perylene		30	40-125	30	40-125	30
8270C DoD PAHs	Water	Chrysene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Dibenz(a,h)anthracene		30	40-125	30	40-125	30
8270C DoD PAHs	Water	Fluoranthene		30	55-115	30	55-115	30
8270C DoD PAHs	Water	Fluorene		30	50-110	30	50-110	30
8270C DoD PAHs	Water	Indeno(1,2,3-cd)pyrene		30	45-125	30	45-125	30
8270C DoD PAHs	Water	2-Methylnaphthalene		30	45-105	30	45-105	30
8270C DoD PAHs	Water	Naphthalene		30	40-100	30	40-100	30
8270C DoD PAHs	Water	Phenanthrene		30	50-115	30	50-115	30
8270C DoD PAHs	Water	Pyrene		30	50-130	30	50-130	30
8270C DoD PAHs	Water	2-Fluorophenol	20-110		-		-	
8270C DoD PAHs	Water	Phenol-d6	10-115		-		-	
8270C DoD PAHs	Water	Nitrobenzene-d5	40-110		-		-	
8270C DoD PAHs	Water	2-Fluorobiphenyl	50-110		-		-	
8270C DoD PAHs	Water	2,4,6-Tribromophenol	40-125		-		-	
8270C DoD PAHs	Water	o-Terphenyl	50-135		-		-	
8270C DoD PAHs	Water	1,4-Dichlorobenzene-d4	-		-		-	
8270C DoD PAHs	Water	Naphthalene-d8	-		-		-	
8270C DoD PAHs	Water	Acenaphthene-d10	-		-		-	
8270C DoD PAHs	Water	Phenanthrene-d10	-		-		-	
8270C DoD PAHs	Water	Chrysene-d12	-		-		-	
8270C DoD PAHs	Water	Perylene-d12	-		-		-	



### QAPP Worksheet #14G Summary of Project Tasks

#### Sampling Tasks

The sampling activities are described in the FSPG&S, Tetra Tech NUS, Inc., 2011.

#### Analysis Tasks

Samples will be processed, prepared and analyzed by Trimatrix Laboratories, Inc. for:

- PAHs (groundwater)
- Michigan 10 Metals (groundwater)
- Toxicity Characteristic Leaching Procedure metals (IDW samples)
- pH (IDW samples)
- Toxicity Characteristic Leaching Procedure volatiles (IDW samples)

#### Quality Control Tasks

The samples will be collected and processed and the waste disposed as documented in field Standard Operating Procedures provided in FSPG&S, Tetra Tech NUS, Inc., 2011. The quality control samples are described in Worksheet #28-1G.

#### Secondary Data

See Worksheet #13.

#### Data Management Tasks

The purpose of data management is to ensure that all of the necessary data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The analytical results from Site evaluation and removal action activities if warranted will be received from the laboratory in an electronic data deliverable format. The data will be incorporated into the existing electronic database.



**QAPP Worksheet #14G Summary of Project Tasks****Documentation and Records**

**Field sample identification** — described in the FSPG&S, Tetra Tech NUS, Inc., 2011.

- **Field documentation** — Field personnel will provide comprehensive documentation covering various aspects of field sampling, field analysis, and sample chain of custody. This documentation consists of a record that allows reconstruction of field events and sample handling to aid in the data review and interpretation process. Documents, records, and information relating to the performance of the field work will be retained in the project file.
- **Laboratory project files** — The laboratory will establish a file for pertinent data. The file will include correspondence, faxed information, phone logs, and chain-of-custody forms. The laboratory will retain project files and data packages for a period not less than 5 years. The USCG and/or its designated representative will retain copies of the analytical data reports.
- **Laboratory logbooks** — Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and to document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory. Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with one stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions. Information regarding the sample, analytical procedures performed, and results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, instrument used, and instrument conditions. Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance with this QAPPA. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.
- **Computer and hard copy storage** — All electronic files and deliverables will be retained by the laboratory for not less than 5 years; hard copy data packages (or electronic copies) will also be retained for not less than 5 years. The USCG and/or its designated representative will retain copies of the analytical data reports.
- **Field data reporting** — Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Field Program Coordinator for adherence to the FSPG&S, Tetra Tech NUS, Inc., 2011 and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and (as necessary) incorporated into the data evaluation process. If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents, and data reductions will be kept in the project file at the Tetra Tech NUS, Inc. office in Pittsburgh, PA.



### QAPP Worksheet #14G Summary of Project Tasks

- **Laboratory data reporting** — Data reports for all parameters will include, at a minimum, the following items:

**Narrative:** Summary of activities that took place during sample analysis, including the following information:

- Laboratory name and address
- Date of sample receipt
- Cross reference of laboratory identification number to contractor sample identification
- Analytical methods used
- Deviations from specified protocol
- Corrective actions taken

Included with the narrative will be any sample handling documents, including field and internal Chain-of-Custody forms, air bills, and shipping tags.

**Analytical Results:** These will be reported according to analysis type and include the following information, as applicable:

- Sample identification
- Laboratory identification
- Date of collection
- Date of receipt
- Date of extraction
- Date of analysis
- Detection limits

Sample results on the report forms will be corrected for dilutions. Soil data will be reported on a dry-weight basis. Unless otherwise specified, all results will be reported uncorrected for blank contamination.

The data associated with Contract Laboratory Program-equivalent reporting will be expanded to include supporting documentation necessary to provide a Contract Laboratory Program-equivalent package. This additional documentation will include, but not be limited to,



**QAPP Worksheet #14G Summary of Project Tasks**

raw data required to recalculate any result, including instrument printouts and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations.

- **Data reporting levels are as follows:**

- **Level 3 — Full Reporting:** Full Contract Laboratory Program or Contract Laboratory Program-type reporting is used for those analyses that, based on the intended data use, require full documentation. Site soil and groundwater samples will also include Level 3 reporting.

**Assessment/Audit Tasks**

Performance and systems audits will be completed in the field and laboratory during the site investigations, as described below and in Worksheets #31 and #32.

1. **Field Audits** — The following field performance and systems audits will be completed during this project.

The Field Program Coordinator will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that the activities are performed according to established procedures. Field performance audits may be performed by the Project Coordinator (or his designee). The auditor(s) will review field reports and communicate concerns to the Project Coordinator and/or Field Program Coordinator, as appropriate.

The number and frequency of field performance audits conducted will be determined independently by the Project Coordinator or Field Program Coordinator. The Project Coordinator (or his/her designee) will conduct field performance audits at a frequency of approximately one per month during field activities. The observations made during field performance audits and any recommended changes/deviations to the field procedures will be recorded and documented.

In addition, the Data Quality Assurance Manager will review the rinsate blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled quality assurance/quality control activities from this QAPPA with actual quality assurance/quality control activities completed will be performed. The Field Program Coordinator and Data Quality Assurance Manager will periodically confirm that work is being performed consistent with this QAPPA and the FSPG&S, Tetra Tech NUS, Inc., 2011.



## QAPP Worksheet #14G Summary of Project Tasks

### 2. Laboratory Audits

Periodic internal laboratory audits are conducted by the Laboratory Quality Assurance Manager. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory quality assurance manual and standard operating procedures. Results of the audits are summarized and issued to each department supervisor, Laboratory Manager, and Laboratory Director. A systems audit of each laboratory is also performed by the Data Quality Assurance Manager to determine whether the procedures implemented by each laboratory comply with the quality assurance manual and standard operating procedures.

As a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification, in addition to the laboratory's internal audits. Audits are usually conducted annually and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

The USCG reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the course of the project, as deemed necessary.

### 3. Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPPA. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

#### a. Field Procedures

If, during fieldwork, a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented by the Field Program Coordinator or a designee will be documented on a Corrective Action Form and reported to the appropriate Field Program Coordinator, Quality Assurance Manager, and Project Coordinator.



### QAPP Worksheet #14G Summary of Project Tasks

Examples of situations that would require corrective actions are provided below:

- Protocols as defined by the QAPPA and/or FSPG&S, Tetra Tech NUS, Inc., 2011 have not been followed.
- Equipment is not in proper working order or is not properly calibrated.
- Quality control requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Project personnel will continuously monitor ongoing work performance as part of daily responsibilities.

#### b. Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the appropriate Project Coordinator and Data Quality Assurance Manager.

Corrective action may be initiated, at a minimum, under the following conditions:

- Protocols as defined by this QAPPA have not been followed.
- Predetermined data acceptance standards are not obtained.
- Equipment is not in proper working order or calibrated.
- Sample and test results are not completely traceable.
- Quality control requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance as part of daily responsibilities. Corrective action is initiated at the point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the Laboratory Quality Assurance Manager and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, reanalysis, cleanup, dilution, matrix modification, or other activities.



**QAPP Worksheet #14G Summary of Project Tasks**

<b>Data Review Tasks</b>
See Worksheets #34, #35, #36G, and #37.



## QAPP Worksheet #15-2G Reference Limits and Evaluation — (Groundwater)

Analyte	CAS #	Method	Performance Standard (ug/L)	Groundwater	
				Laboratory RL (ug/L)	Laboratory MDL (ug/L)
Metals					
ARSENIC	7440-38-2	6020A	10 (MCL)	5	0.381
BARIUM	7440-39-3	6020A	670 (MI-GSI)	2	0.589
CADMIUM	7440-43-9	6020A	2.5 (MI-GSI)	0.2	0.0548
CHROMIUM	7440-47-3	6020A	11 (MCL for Cr VI)	2	0.455
COPPER	7440-50-8	6020A	13 (MI-GSI)	2	0.708
LEAD	7439-92-1	6020A	4 (MI-DWC)	1	0.215
MERCURY	7439-97-6	7470A	2 (MCL)	0.2	0.0613
SELENIUM	7782-49-2	6020A	5 (MI-GSI)	5	0.821
SILVER	7440-22-4	6020A	0.2 (MI-GSI)	0.2	0.0612
ZINC	7440-66-6	6020A	170 (MI-GSI)	10	2.91
PAHs					
2-METHYLNAPHTHALENE	91-57-6	8270C	260 (MIDWC)	0.5	0.0207
ACENAPHTHENE	83-32-9	8270C	19 (MI-GSI)	0.5	0.0195
ACENAPHTHYLENE	208-96-8	8270C	52 (MIDWC)	0.5	0.0332
ANTHRACENE	120-12-7	8270C	43 (MIDWC)	0.5	0.0347
BENZO (A) ANTHRACENE	56-55-3	8270C	2.1 (MIDWC)	0.5	0.0253
BENZO (A) PYRENE	50-32-8	8270C	5 (MCL)	0.5	0.0279
BENZO (B) FLUOROANTHENE	205-99-2	8270C	1.5 (MIDWC)	0.5	0.0289
BENZO (G,H,I) PERYLENE	191-24-2	8270C	1 (MIDWC-DL)	0.5	0.0552
BENZO (K) FLUOROANTHENE	207-08-9	8270C	1 (MIDWC-DL)	0.5	0.0489
CHRYSENE	218-01-9	8270C	1.6 (MIDWC)	0.5	0.0221
DIBENZO (A,H)-ANTHRACENE	53-70-3	8270C	2 (MIDWC-DL)	0.5	0.0532
FLUORANTHENE	206-44-0	8270C	1.6 (MI-GSI)	0.5	0.0402
FLUORENE	86-73-7	8270C	12 (MI-GSI)	0.5	0.0194
INDENO (1,2,3-CD)-PYRENE	193-39-5	8270C	2 (MIDWC-DL)	0.5	0.0418
NAPHTHALENE	91-20-3	8270C	13 (MI-GSI)	0.5	0.0215
PHENANTHRENE	85-01-8	8270C	2.4 (MI-GSI)	0.5	0.0252
PYRENE	129-00-0	8270C	140 (MIDWC)	0.5	0.0255

## Notes:

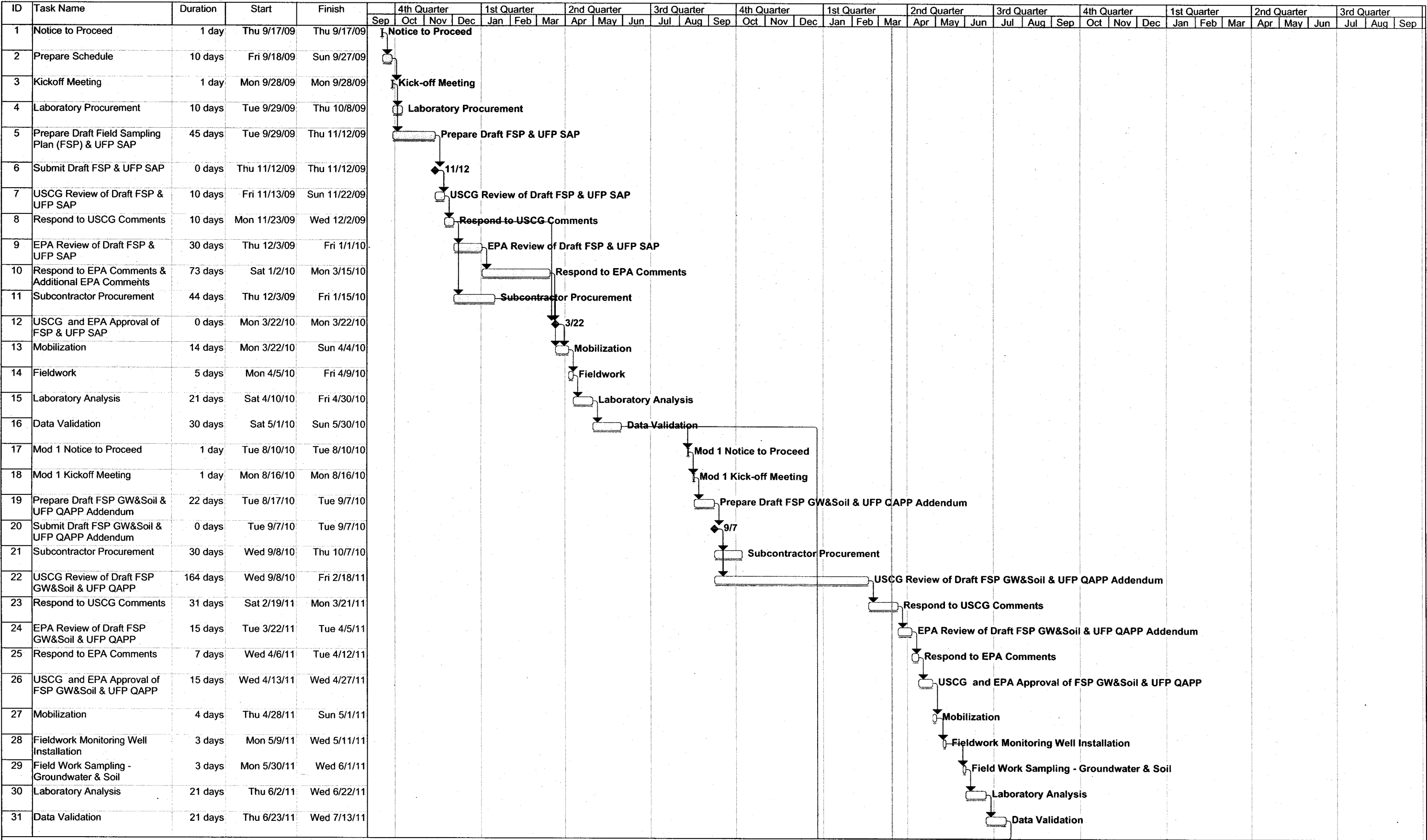
CAS #	Chemical Abstract Service Number
PAH	Polynuclear Aromatic Hydrocarbons
RL	Reporting limit.
ug/L	Micrograms per liter.
MDL	Method Detection Limit
MID-GSI	Michigan DNRE Groundwater Surface Water Interface Protection Criteria
MIDWC	Michigan DNRE Drinking Water Criteria
MIDWC-DL	Michigan DNRE Drinking Water Criteria, based on laboratory detection limit.
MCL	Maximum Contaminant Level



**QAPP Worksheet #16G Project Schedule/Timeline**

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Quality Assurance Project Plan Addendum	Tetra Tech NUS, Inc.	August 2010	April 2011	Yes	March 2011 (Draft) April 2011 (Final)
Field Sampling Plan – Groundwater and Soil	Tetra Tech NUS, Inc.	August 2010	April 2011	Yes	March 2011 (Draft) April 2011 (Final)
Mobilization (Site preparation activities)	Tetra Tech NUS, Inc.	May 2011	May 2011	No	Not Applicable
Site Investigation (Monitoring wells, collect samples)	Tetra Tech NUS, Inc.	May 2011	May 2011	No	Not Applicable
Site Assessment Report/HHRA	Tetra Tech NUS, Inc.	December 2010	September 2011	Yes	August 2011 (Draft) September 2011 (Final) (to USCG)
Engineering Evaluation/ Cost Analysis	Tetra Tech NUS, Inc.	August 2011	November 2011	Yes	September 2011 (Draft) (to USCG) October 2011 (Draft) (to EPA) November 2011 (Final)
Remedial Action Work Plan	Tetra Tech NUS, Inc.	November 2011	March 2012	Yes	January 2012 (Draft) March 2012 (Final)





Project: Detroit Atwater  
CERCLA Process  
Date: Tue 3/15/11

Task

Split

Progress

Milestone

Summary

Rolled Up Task

Rolled Up Split

Rolled Up Milestone

Rolled Up Progress

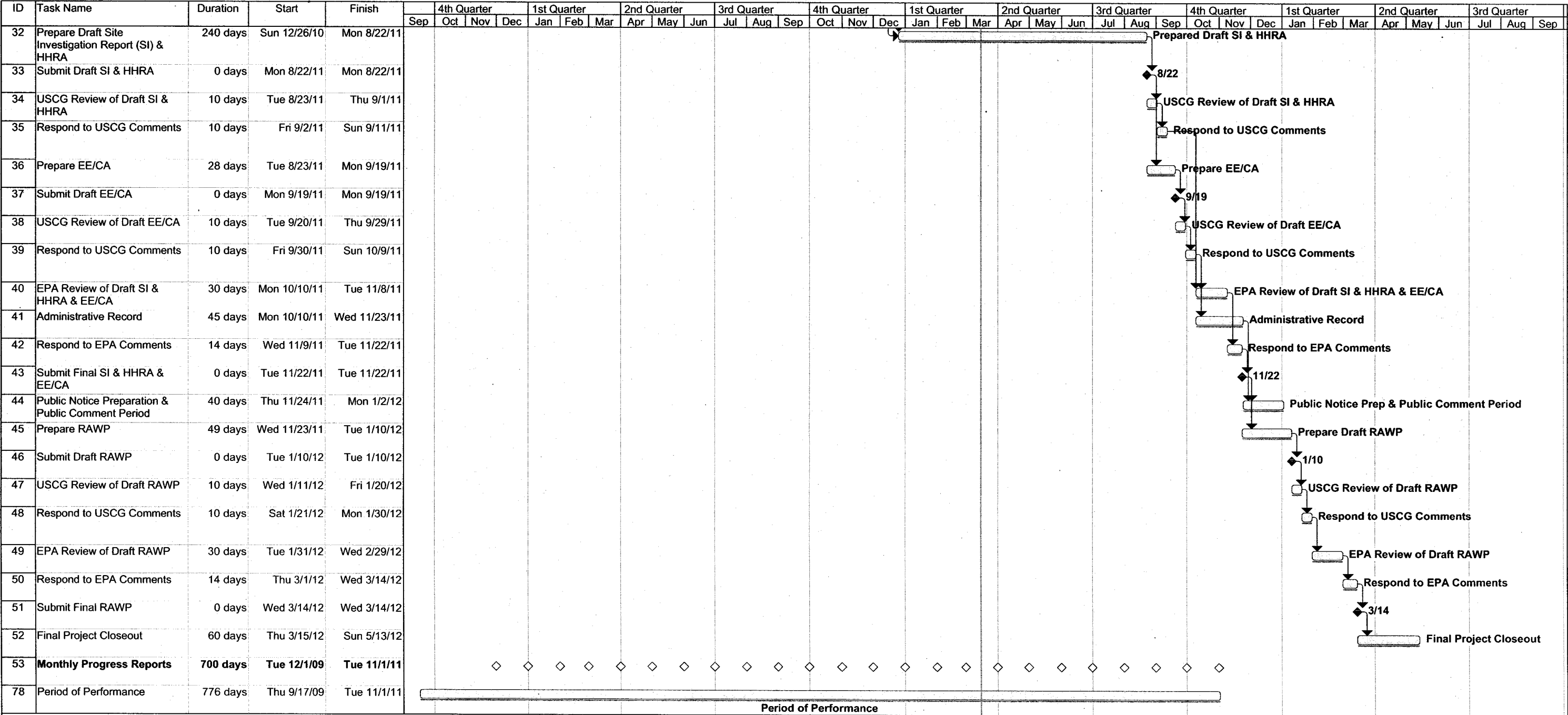
External Tasks

Project Summary

External Milestone

Deadline





Project: Detroit Atwater  
CERCLA Process  
Date: Tue 3/15/11

Task		Milestone		Rolled Up Split		External Tasks		Deadline	
Split		Summary		Rolled Up Milestone		Project Summary			
Progress		Rolled Up Task		Rolled Up Progress		External Milestone			



**QAPP Worksheet #17G Sampling Design and Rationale****Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):**

Details of the Site evaluation sampling program are provided in the FSPG&S, Tetra Tech NUS, Inc., 2011.

**Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):**

Based on previous investigations groundwater samples will be collected to evaluate the potential for PAH and metals impact onsite. The estimated number of samples, including type and frequency of quality control samples, is provided in Worksheet #18G and in the FSPG&S, Tetra Tech NUS, Inc., 2011. Based on a NOAA stream gauge in Ft. Wayne, MI the Detroit River can vary by 2 to 5 feet throughout the year, with the highest observations in June and July (NOAA, 2011). Future groundwater sampling events will take this into consideration.

Three environmental site assessments were conducted at the Atwater site. The first was a Phase I and II Environmental Site Assessment for Detroit Atwater conducted by Tetra Tech NUS, Inc. December 2002. This assessment identified four potential areas of concern: possible asbestos – containing building materials, the AST, oil and gas storage area, and the filled – in slip area. Tetra Tech NUS, Inc. (TtNUS) then conducted a Phase II investigation where seven soil samples were collected. Samples were analyzed for volatile organic compounds (VOCs), semivolatile organic compounds, and RCRA metals. Results identified a few semivolatiles and metals exceeding MDEQ Michigan residential soil criteria results (TtNUS, 2002).

The second a Phase II Environmental Site Assessment prepared for the Economical Development Corporation of the City of Detroit, conducted by Enviro Matrix in July 2006. The Phase I indicated three potential recognized environmental conditions (RECs) exist at the site. The RECs identified were: Fill of unknown origin may have been brought into the parcel, the property has been used by the USCG and other unknown uses dating back to the late 1800's, and heavy industrial activities in the area. Subsequently, Enviro Matrix conducted a Phase II site investigation five soil borings were advanced and six soil samples were collected for PAHs, metals (Michigan 10) and one location was analyzed for VOCs. This investigation identified elevated PAHs and metals greater than the most restrictive MDEQ (Enviro Matrix, 2006).

The third Site Investigation was conducted by Tetra Tech NUS April 2010. During the most recent field investigation four borings (DASB102, DASB106, DASB110, and DASB112) were advanced to 15 feet bgs and left open overnight to monitor the accumulation of groundwater. Three of the four borings were observed to have accumulated water. Based on these observations, four permanent monitoring wells will be installed and sampled.



**QAPP Worksheet #18G Sampling Locations and Methods/Standard Operating Procedure Requirements**

Sampling Location/Identification Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates) <sup>1</sup>	Sampling Standard Operating Procedure Reference Number <sup>2</sup>	Rationale for Sampling Location
DAMW-01 through DAMW-04	Aqueous	4 grab samples collected from permanent monitoring wells	PAHs and Metals	Normal	4 + 1 field duplicate	FSPG&S, Tetra Tech NUS, Inc., 2011.	PAH and metals impact to groundwater; FSPG&S, Tetra Tech NUS, Inc., 2011.
DASS102A-, DASS104A-, DASS106A-, DASS109A-, DASS110A-, DASS111A-, DASS112A- and DASS115A-XXXX	Soil	8 grab samples: from 0-2' bgs	PAHs and Mercury	Normal	8 + 1 field duplicate	FSPG&S, Tetra Tech NUS, Inc., 2011.	Vertical and horizontal delineation of PAH and metal impacts: FSPG&S, Tetra Tech NUS, Inc., 2011.
DASB102A-, DASB104A-, DASB106A-, DASB109A-, DASB110A-, DASB111A-, DASB112A- and DASB115A-XXXX	Soil	8 grab samples: from a subsurface depth to be determined in the field	PAHs and Mercury	Normal	8 + 1 field duplicate	FSPG&S, Tetra Tech NUS, Inc., 2011.	Vertical and horizontal delineation of PAH and metal impacts: FSPG&S, Tetra Tech NUS, Inc., 2011.

**Notes:**

1 One blind duplicate sample will be collected for every 10 samples.

2 Reference number from QAPP Worksheet #21.



## QAPP Worksheet #19G Analytical Standard Operating Procedure Requirements (Sample Containers, Preservation, and Holding Times)

Parameter	Analytical and Preparation Method/SOP Reference	Method	Bottle Type	Preservation	Holding Time
<b>Groundwater</b>					
Metals (Michigan 10)	GR-01-129, 6020A; GR-01-148; 3020A; GR-01-123, GR-01-140 245.1 7470A	6020A/3020A 7470A	One 500 mL Plastic bottle	pH < 2 with HNO <sub>3</sub> , Cool to 4°C±2°C	180 days to analysis; mercury 28 days to analysis
PAHs	GR-04-103, 8270C; GR-09-101, 3510C	8270C/3510C	Two 1000 mL amber glass jar	Cool to 4°C±2°C	7 days to extraction/40 days from extract to analysis
<b>IDW</b>					
TCLP/Metals	GR-01-119, 1311; GR-01-147, 3010A; GR-01-100, 6010C, GR-01-140, 7470A Prep; GR-01-123, 7470A	1311/6010C/7470A	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	14 days to TCLP extraction; 180 days to analysis; mercury 28 days to analysis
TCLP/Volatile Organic Compounds	GR-01-119, 1311; GR-04-104, 8260B	1311/8260B	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	14 days to TCLP extraction; 14 days to analysis
pH	GR-07-113, 9045C	9045C	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	7 days to analysis

**Notes:**

°C Degrees Celsius.

SOP Standard Operating Procedure.



## QAPP Worksheet #20G Sample Quantities and Control Frequencies

	Laboratory	Analytical and Preparation Standard Operating Procedure <sup>1</sup>	Estimated Environ. Sample Quality	Field Quality Control Analyses						Laboratory Quality Control Sample						Total
				Trip Blank		Rinsate Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Laboratory Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Groundwater Sampling																
PAHs	Trimatrix	GR-04-103 and GR-09-101	4	NA	--	1/day or 1/20, whichever is greater	0	1/10	1	1/20	1	1/20	1	NA	NA	3
Metals (Michigan 10)	Trimatrix	GR-01-129, GR-01-123, and GR-01-148	4	NA	--	1/day or 1/20, whichever is greater	0	1/10	1	1/20	1	1/20	1	NA	NA	3

**Notes:**

1. See Worksheet #23 for Standard Operating Procedure title, revision number, date details.

A. Number of samples is estimated. Number of samples collected will be based on Site evaluation results, field observations and final excavation sizes, etc.

1/day One per day or one per 20 samples, whichever is more frequent. Rinseate blanks not required when dedicated sampling equipment is used.

Freq Frequency.

NA Not Applicable.

No. Number.

SOP Standard Operating Procedure.



**QAPP Worksheet #21G Field Sampling Standard Operating Procedure References**

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Soil Sampling Procedures</i>	Tetra Tech NUS, Inc.	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedures for soil sampling and the required equipment
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Field Equipment Decontamination Procedures</i>	Tetra Tech NUS, Inc.	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedure for field equipment cleaning and decontamination
FSPG&S, Tetra Tech NUS, Inc., 2011 and QAPP Worksheet #27	FSPG&S, Tetra Tech NUS, Inc., 2011 and QAPP Worksheet #27— <i>Field Samples Handling, Packing, and Shipping Procedures</i>	Tetra Tech NUS, Inc.	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes field sample handling, packaging, and shipping procedures
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Borehole and sample logging</i>	Tetra Tech NUS, Inc	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedures for soil logging and the required equipment
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Direct Push Technology</i>	Tetra Tech NUS, Inc	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedures for Direct Push Technology and the required equipment
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Groundwater Monitoring Well Installation</i>	Tetra Tech NUS, Inc	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedures for monitoring well installation.
FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011 — <i>Groundwater Sample Acquisition and Onsite Water Quality Testing</i>	Tetra Tech NUS, Inc	See FSPG&S, Tetra Tech NUS, Inc., 2011 for specific equipment needs	N	Describes the procedures for groundwater sampling and the required equipment.



**QAPP Worksheet #22G Field Equipment Calibration, Maintenance, Testing, and Inspection**

Field Equipment	Calibration Activity/ Frequency	Maintenance Activity	Testing Activity	Inspection Activity	Acceptance Criteria	Corrective Action	Responsible Person	Standard Operating Procedure Reference <sup>1</sup>
PID	FSPG&S, Tetra Tech NUS, Inc., 2011	As required by manufacturer specifications	Screen for VOCs	Check all sensors, cable and check battery charge	FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011	Tetra Tech NUS, Inc. Field Staff	FSPG&S, Tetra Tech NUS, Inc., 2011
Field Water Quality Meter (s)	FSPG&S, Tetra Tech NUS, Inc., 2011	As required by manufacturer specifications	Collect groundwater quality field parameters during purging and sampling	Check all sensors, cable and check battery charge	FSPG&S, Tetra Tech NUS, Inc., 2011	FSPG&S, Tetra Tech NUS, Inc., 2011	Tetra Tech NUS, Inc. Field Staff	FSPG&S, Tetra Tech NUS, Inc., 2011

**Notes:**

<sup>1</sup> Standard Operating Procedures are found in the FSPG&S, Tetra Tech NUS, Inc., 2011



## QAPP Worksheet #23G Analytical Standard Operating Procedure References

Standard Operating Procedure Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
GR-01-129	Inductively Coupled Plasma-Mass Spectrometry, 03/10/10 -- rev. 3.9 6020A	Definitive	Metals - Groundwater	Perkin Elmer ELAN ICP-MS 6000 6100	TriMatrix	N
GR-01-123	Mercury by Semi-Automated Cold Vapor Atomic Absorption 04/15/10 -- rev. 5.7, 7470A/7471A	Definitive	Mercury - Groundwater	PSA Millenium Cold Vapor Mercury Analyzer	TriMatrix	N
GR-04-103	Base/Neutral/Acid Compounds by Gas Chromatography/ Mass Spectrometry, 02/20/09 -- rev. 5.5, 8270C	Definitive	PAHs - Groundwater	Agilent GC 6890 5973N MSD or Agilent GC 6890 5975N MSD	TriMatrix	N
GR-01-148	Block Digestion of Aqueous samples and Extracts for Total / Dissolved Metals by ICP_MS 04/09/10 rev 0.3, 3020A	Definitive	Metals - Groundwater	Environmental Express Hot Block	TriMatrix	N
GR-01-140	Block Digestion of Mercury in Water, Wastewater, and Aqueous Waste 01/22/09 rev 0.4, 245.1, 7470A	Definitive	Mercury - Groundwater	Environmental Express Hot Block	TriMatrix	N
GR-09-101	Extraction of BNA Semi-Volatiles in Water, 04/16/10 - - rev. 3.3, 3510C	Definitive	PAHs - Groundwater	NA	TriMatrix	N



## QAPP Worksheet #27G Sample Custody Requirements

### Sample Handling and Custody Requirements

Field and laboratory personnel will, at all times, be aware of the need to maintain all samples, whether in the field or in the laboratory, under strict chain of custody and in a manner to retain physical properties and chemical composition. This Worksheet details sample handling and custody requirements from collection to ultimate disposal.

### Sample Handling (Sample Packaging, Shipping Containers and Sample Shipment, Sample Custody)

Sample packaging and shipment procedures are designed so that the samples will arrive at the laboratory, with the chain-of-custody, intact.

Samples will be packaged for shipment as outlined below:

- Securely affix the sample label to the container with clear packing tape.
- Check the cap on the sample container to confirm that it is properly sealed.
- Wrap the sample container cap with clear packing tape to prevent the label from becoming loose.
- Complete the chain-of-custody form with the required sampling information and confirm that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain-of-custody form prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.
- Place chain-of-custody forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock and secure with duct tape.
- Wrap strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address. Cover the labels with clear plastic tape.
- Place a signed custody seal over the sample cooler lid.

Samples will be packaged by the field personnel and transported as low-concentration environmental samples. Samples will be hand delivered or delivered by an express carrier within 48 hours of the time of collection.



## QAPP Worksheet #27G Sample Custody Requirements

### Sample Handling (Sample Packaging, Shipping Containers and Sample Shipment, Sample Custody) Continued

Shipments will be accompanied by the chain-of-custody form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the chain-of-custody form as long as the forms are sealed inside the sample cooler, and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratory. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the likelihood of container breakage.

Additional procedures for packing, handling, and shipping environmental samples are presented in the FSPG&S, Tetra Tech NUS, Inc., 2011.

### Field Custody Procedures

The objective of field sample custody is to protect samples from tampering from the time of sample collection through time of transport to the analytical laboratory. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field chain-of-custody forms.

Field logbooks will provide the means of recording the data collecting activities that are performed. As such, entries will be described in as much detail as possible so that persons going to the Site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- End date



## QAPP Worksheet #27G Sample Custody Requirements

### Field Custody Procedures Continued

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather conditions, names of all sampling team members present, level of personal protection being used, and signature of the person making the entry will be provided. The names of visitors to the Site and field sampling or investigation team personnel, as well as the purpose of their visit, will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, with no erasures. If an incorrect entry is made, the information will be crossed out with one strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded. The number of the photographs taken, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in FSPG&S, Tetra Tech NUS, Inc., 2011. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

### Sample Labels

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- Project name
- Date collected
- Time collected
- Location
- Sampler
- Analysis to be performed
- Preservative
- Sample number

Sample labels will be completed for each sample using waterproof ink. The labels will include the information listed above. The completed sample labels will be affixed to each sample bottle and covered with clear tape.



## QAPP Worksheet #27G Sample Custody Requirements

### Chain of Custody Record

Completed chain-of-custody forms will be required for all samples to be analyzed. Chain-of-custody forms will be initiated by the sampling crew in the field. The chain-of-custody forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any), and analyses required. The original chain-of-custody form will accompany the samples to the laboratory. Copies of the chain-of-custody will be made prior to shipment (or multiple copy forms will be used) for field documentation. The chain-of-custody forms will remain with the samples at all times. The samples and signed chain-of-custody forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express), hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Whenever samples are split with a government agency or other party, a separate chain of custody will be prepared for those samples and marked to identify the party with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

### Laboratory Custody Procedures

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain-of-custody form will accompany all samples requiring laboratory analysis. The laboratory will use chain-of-custody guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

Immediately upon sample receipt, the laboratory sample custodian will verify the integrity of the cooler seal, open the cooler, and compare the contents against the field chain of custody. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means, Tetra Tech NUS, Inc. staff will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.



## QAPP Worksheet #28-1G Quality Control Samples — 6020A Metals (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	6020A/GR-01-129	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	Metals	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
Field Duplicate	One per 10 field samples of similar matrix	RPD < 30%	Qualify data as needed	TtNUS personnel	Precision — overall	RPD < 30%
Method Blanks	1/Preparation Batch (20 Samples)	No target analytes > ½ RL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply B flag to all affected sample results	Metals Analyst	Accuracy/bias contamination	No target analytes > ½ RL.
Equipment Blanks	One per day or one per 20 field samples, whichever is greater	< RL	Qualify data as needed	TtNUS personnel	Accuracy/bias contamination	< RL
Calibration Verification Standards	Before samples are analyzed, after every 10 samples, and at the end of analytical sequence	%R (90-110%)	Reanalysis of all samples bracketed by out of control CCVs	Metals Analyst	Accuracy/bias contamination	%R (90-110%)



## QAPP Worksheet #28-1G Quality Control Samples — 6020A Metals (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	6020A/GR-01-129	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	Metals	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
MS/MSD	1/Preparation Batch (20 Samples)	80%-120% Recovery RPD < 20% between MS and MSD	Check for errors in calculations and spike preparation. Check unspiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, then matrix effects are the likely cause. Qualify failing analytes as estimated.	Metals Analyst	Accuracy/Precision	80%-120% Recovery RPD < 20% between MS and MSD
LCS	1/Preparation Batch (20 Samples))	80%-120% Recovery	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply qualifier to all affected sample results.	Metals Analyst	Accuracy	80%-120% Recovery
Post Digestion Spike	1/Preparation Batch (20 Samples)	Recovery within 75- 125% of expected value	Apply J-flag to associated sample result. Not necessary if analyte concentration < 100 x MDL.	Metals Analyst	Accuracy	Recovery within 75-125% of expected value



## QAPP Worksheet #28-1G Quality Control Samples — 6020A Metals (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	6020A/GR-01-129	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	Metals	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
Internal Standards (IS)	Every Sample	IS intensity within 70- 120% of the intensity of the IS in the ICAL	Reanalyze sample at 5-fold dilution.	Metals Analyst	Accuracy	IS intensity within 70-120% of the intensity of the IS in the ICAL

**Notes:** A preparation batch is defined as no more than 20 analytical samples including field samples, blanks, matrix spike/matrix spike duplicates, and laboratory control samples/laboratory control sample duplicates.

LCS Laboratory control sample.

No. Number.

RL Reporting limit.

RPD Relative percent difference.

SOP Standard operating procedure.

%D Percent difference.

%R Percent recovery.



## QAPP Worksheet #28-1G Quality Control Samples — Mercury (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	SW-846 7470AGR-01-123	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	Mercury	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S, Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
Field Duplicate	One per 10 field samples of similar matrix	RPD < 30%	Qualify data as needed	TtNUS personnel	Precision — overall	RPD < 30%
Method Blanks	1/Preparation Batch (20 Samples)	No target analytes > ½ RL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply B flag to all affected sample results	Metals Analyst	Accuracy/bias contamination	No target analytes > ½ RL.
Equipment Blanks	One per day or one per 20 field samples, whichever is greater	< RL	Qualify data as needed	TtNUS personnel	Accuracy/bias contamination	< RL
Calibration Verification Standards	Before samples are analyzed, after every 10 samples, and at the end of analytical sequence	%R (80-120%)	Reanalysis of all samples bracketed by out of control CCVs	Metals Analyst	Accuracy/bias contamination	%R (80-120%)



## QAPP Worksheet #28-1G Quality Control Samples — Mercury (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	SW-846 7470AGR-01-123	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	Mercury	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S, Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
MS/MSD	1/Preparation Batch (20 Samples)	80%-120% Recovery RPD < 20% between MS and MSD	Check for errors in calculations and spike preparation. Check unspiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, then matrix effects are the likely cause. Qualify failing analytes as estimated.	Metals Analyst	Accuracy/Precision	80%-120% Recovery RPD < 20% between MS and MSD
LCS	1/Preparation Batch (20 Samples))	80%-120% Recovery	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply qualifier to all affected sample results.	Metals Analyst	Accuracy	80%-120% Recovery



**Notes:** A preparation batch is defined as no more than 20 analytical samples including field samples, blanks, matrix spike/matrix spike duplicates, and laboratory control samples/laboratory control sample duplicates.

LCS Laboratory control sample.

No. Number.

RL Reporting limit.

RPD Relative percent difference.

SOP Standard operating procedure.

%D Percent difference.

%R Percent recovery.



## QAPP Worksheet #28-1G Quality Control Samples — 8270C PAH (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	SW-846 8270C/ GR-04-103	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	PAHs	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S, Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
Field Duplicate	One per 10 field samples of similar matrix	RPD < 30%	Qualify data as needed	TtNUS personnel	Precision — overall	RPD < 30%
Method Blanks	1/Preparation Batch (20 Samples)	No target analytes > ½ RL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply B flag to all affected sample results	GC/MS Analyst	Accuracy/bias contamination	No target analytes > ½ RL.
Equipment Blanks	One per day or one per 20 field samples, whichever is greater	< RL	Qualify data as needed	TtNUS personnel	Accuracy/bias contamination	< RL



## QAPP Worksheet #28-1G Quality Control Samples — 8270C PAH (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	SW-846 8270C/ GR-04-103	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	PAHs	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S, Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
Calibration Verification Standards	Analyze a standard at the beginning of each 12-hour shift after decafluorotriphenylph osphine (DFTPP) tune.	%R (80-120%)	Check standard preparation, perform any necessary instrument maintenance and repeat .If CCV fails again, then repeat initial calibration. Reanalyze all samples with failed calibration verification.	GC/MS Analyst	Accuracy/bias contamination	%R (80-120%)
MS/MSD	1/Preparation Batch (20 Samples)	Table G-6 QSM Version 4.1 (see attached Appendix A) %RPD <30	Check for errors in calculations and spike preparation. Check unspiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, then matrix effects are the likely cause. Qualify failing analytes as estimated.	GC/MS Analyst	Accuracy/Precision	Table G-6 QSM Version 4.1 (see attached Appendix A) %RPD <30



## QAPP Worksheet #28-1G Quality Control Samples — 8270C PAH (Groundwater)

<b>Matrix</b>	Groundwater	<b>Analytical Method/ SOP Reference</b>	SW-846 8270C/ GR-04-103	<b>No. of Sample Locations</b>	Numerous	
<b>Analytical Group</b>	PAHs	<b>Sampler's Name</b>	Not Applicable			
<b>Concentration Level</b>	All	<b>Field Sampling Organization</b>	TtNUS			
<b>Sampling Standard Operating Procedure</b>	FSPG&S, Tetra Tech NUS, Inc., 2011	<b>Analytical Organization</b>	TriMatrix			
<b>Quality Control Sample</b>	<b>Frequency/Number</b>	<b>Method/Standard Operating Procedures Quality Control Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator</b>	<b>Measurement Performance Criteria</b>
LCS	1/Preparation Batch (20 Samples))	Table G-6 QSM Version 4.1 (see attached Appendix A)	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, then apply qualifier to all affected sample results.	GC/MS Analyst	Accuracy	Table G-6 QSM Version 4.1 (see attached Appendix A)
Surrogates	3 per sample: Nitrobenzene-d5 2-Fluorobiphenyl ortho-Terphenyl	40-110% 50-110% 50-135%	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy/Bias	40-110% 50-110% 50-135%
Internal Standards	6 per sample: 1,4-Dichlorobenzene- d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time +/- 30 seconds from RT of the midpoint calibration initial calibration standard, and the area within -50% to +100 % of initial calibration midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, then reanalyze affected sample if sufficient sample is available. Qualify data as needed.	GC/MS Analyst	Accuracy/Bias	Retention time +/- 30 seconds from RT of the midpoint calibration initial calibration standard, and the area within -50% to +100 % of initial calibration midpoint standard.



**Notes:** A preparation batch is defined as no more than 20 analytical samples including field samples, blanks, matrix spike/matrix spike duplicates, and laboratory control samples/laboratory control sample duplicates.

LCS Laboratory control sample.

No. Number.

RL Reporting limit.

RPD Relative percent difference.

SOP Standard operating procedure.

%D Percent difference.

%R Percent recovery.

QSM DoD Quality Systems Manual, Version 4.1

PAH Polynuclear Aromatic Hydrocarbons

GC/MS Gas Chromatograph/Mass Spectrometer



## Appendix A - 8270C PAH LCS/MS/MSD Control Limits

Analysis	Matrix	Analyte	Sur	Dup	MS	MSRPD	BS	BSRPD
8270C DoD PAHs	Water	Acenaphthene		30	45-110	30	45-110	30
8270C DoD PAHs	Water	Acenaphthylene		30	50-105	30	50-105	30
8270C DoD PAHs	Water	Anthracene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(a)anthracene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(a)pyrene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Benzo(b)fluoranthene		30	45-120	30	45-120	30
8270C DoD PAHs	Water	Benzo(k)fluoranthene		30	45-125	30	45-125	30
8270C DoD PAHs	Water	Benzo(g,h,i)perylene		30	40-125	30	40-125	30
8270C DoD PAHs	Water	Chrysene		30	55-110	30	55-110	30
8270C DoD PAHs	Water	Dibenz(a,h)anthracene		30	40-125	30	40-125	30
8270C DoD PAHs	Water	Fluoranthene		30	55-115	30	55-115	30
8270C DoD PAHs	Water	Fluorene		30	50-110	30	50-110	30
8270C DoD PAHs	Water	Indeno(1,2,3-cd)pyrene		30	45-125	30	45-125	30
8270C DoD PAHs	Water	2-Methylnaphthalene		30	45-105	30	45-105	30
8270C DoD PAHs	Water	Naphthalene		30	40-100	30	40-100	30
8270C DoD PAHs	Water	Phenanthrene		30	50-115	30	50-115	30
8270C DoD PAHs	Water	Pyrene		30	50-130	30	50-130	30
8270C DoD PAHs	Water	2-Fluorophenol	20-110		-		-	
8270C DoD PAHs	Water	Phenol-d6	10-115		-		-	
8270C DoD PAHs	Water	Nitrobenzene-d5	40-110		-		-	
8270C DoD PAHs	Water	2-Fluorobiphenyl	50-110		-		-	
8270C DoD PAHs	Water	2,4,6-Tribromophenol	40-125		-		-	
8270C DoD PAHs	Water	o-Terphenyl	50-135		-		-	
8270C DoD PAHs	Water	1,4-Dichlorobenzene-d4	-		-		-	
8270C DoD PAHs	Water	Naphthalene-d8	-		-		-	
8270C DoD PAHs	Water	Acenaphthene-d10	-		-		-	
8270C DoD PAHs	Water	Phenanthrene-d10	-		-		-	
8270C DoD PAHs	Water	Chrysene-d12	-		-		-	
8270C DoD PAHs	Water	Perylene-d12	-		-		-	



**QAPP Worksheet #29G Project Documents and Records**

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
<ul style="list-style-type: none"> <li>- Field Notes</li> <li>- Sampling Logs</li> <li>- Chain-of-Custody Records</li> <li>- Air Bills</li> <li>- Custody Seals</li> <li>- Purge logs</li> </ul>	<ul style="list-style-type: none"> <li>- Equipment Calibration Logs</li> <li>- Field Data Records</li> <li>- Field Instrument Maintenance Logs</li> </ul>	<ul style="list-style-type: none"> <li>- Sample Receipt, Custody and Tracking Records</li> <li>- Standard Traceability Logs</li> <li>- Equipment Calibration Logs</li> <li>- Sample Prep Logs</li> <li>- Run Logs</li> <li>- Equipment Maintenance, Testing, and Inspection Logs</li> <li>- Corrective Action Forms</li> <li>- Reported Field Sample Results</li> <li>- Reported Results for Standards, Quality Control Checks and Quality Control Samples</li> <li>- Instrument Printouts (raw data) for Field Samples, Standards, Quality Control Checks and Quality Control Samples</li> <li>- Data Package Completeness Checklists</li> <li>- Sample Disposal Records</li> <li>- Extraction/Cleanup Records</li> <li>- Raw Data (stored on disk or CD-R)</li> <li>- Analytical Reports</li> </ul>	<ul style="list-style-type: none"> <li>- Data Validation Checklists</li> <li>- Data Quality Assessments</li> </ul>	<ul style="list-style-type: none"> <li>- Health and Safety Plan</li> <li>- QAPP Tetra Tech NUS, Inc., 2010</li> <li>- FSPG&amp;S, Tetra Tech NUS, Inc., 2011</li> </ul>



**QAPP Worksheet #30G Analytical Services**

Matrix	Analytical Group	Concentration Level	Analytical Standard Operating Procedures	Data Package Turnaround Time (calendar days)	Laboratory/Organization (name and address, contact person and telephone number)	Backup Laboratory/Organization (name and address, contact person and telephone number)
Soil	PAHs and Metals (Michigan 10)	All	See Worksheet #23G	21 calendar days	TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court SE Grand Rapids, MI 49512 Gary Wood 616.940.4206	Not Applicable
Groundwater	PAHs and Metals (Michigan 10)	All	See Worksheet #23G	21 calendar days	TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court SE Grand Rapids, MI 49512 Gary Wood 616.940.4206	Not Applicable



**QAPP Worksheet #36G Validation (Steps IIa and IIb) Summary**

Step IIa/IIb	Matrix	Analytical Group	Data Purpose	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
Steps IIa and IIb	Groundwater	Metals	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA <i>Contract Laboratory Program National Functional Guidelines for Inorganic Data Review</i> , EPA 540-R-04-004, October 2004; SW-846 method-specific criteria,, DOD QSM, laboratory SOPs and control limits, QAPP Worksheet 12 and 28, and professional judgment.	Tetra Tech

During the data review process, laboratory data are evaluated against the requirements outlined Worksheet #36. Based on this evaluation, qualifiers may be added, removed, or modified by the data reviewer. Results are flagged with the following qualifiers in accordance with the USEPA National Functional Guidelines:

- Validation Qualifiers

- J The analyte was positively identified; however, the associated numerical value is an estimated concentration only.
- UJ The analyte was not detected above the reported sample detection limit. However, the reported limit is approximate and may or may not represent the actual limit of detection (LOD).
- U The analyte was analyzed for but not detected. The associated value is the limit of detection (LOD).
- UR Nondetected sample results are rejected.
- R Positive sample results are rejected.



The "R" and "UR" flags mean that the associated result is unusable. In other words, due to significant quality control problems, the analysis is invalid and provides no usable information as to whether the analyte is present or not. "R" and "UR" results should not appear on data tables because these data points are not considered reliable.

QAPP Worksheet #36G-A Validation Data Qualification Summary				
Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Holding Time and Sample Preservation	Each Environmental Sample	180 days from collection to analysis	Analysis > 180 days	Non-detect = UR, Detect = J
Tune	Prior to each initial calibration	Tune must be performed; mass calibration must be within 0.1 amu for any isotope; for stability, RSD ≤5%	>0.1 amu	Non-detect = UJ, Detect = J
			Tune not performed.	Non-detect = UR, Detect = R
Initial Calibration - Minimum of a five point calibration for all target analytes.	Initial Calibration performed daily prior to sample analysis.	Correlation Coefficient: $r \geq 0.995$ .	Correlation Coefficient: $r \geq 0.995$ .	Non-detect = UJ, Detect = J
Second-source calibration verification	Immediately after calibration curve	All analytes within +/- 10% of true value	75% to 89%	Non-detect = UJ, Detect = J
			111% to 125%	Non-detect = No action, Detect = J
			<75%	Non-detect = UR, Detect = J



**QAPP Worksheet #36G-A Validation Data Qualification Summary**

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
			>125%	Non-detect = No action Detect = J
Initial calibration blank and Continuing calibration blank (ICB)/(CCB)	Immediately after calibration curve and prior to sample analysis, after every 10 samples, and at the end of analytical sequence.	No analytes detected >1/2 Limit of Quantitation (LOQ)	Detected sample results <LOQ and <blank action level (BAL)	U at the LOQ
			Detected sample results >LOQ and <blank action level (BAL)	U at detected sample concentration
Initial calibration verification (ICV) and Continuing calibration verification	Immediately after calibration curve and prior to sample analysis, after every 10 samples, and at the end of analytical sequence.	All analytes within +/- 10% of true value	75% to 89%	Non-detect = UJ, Detect = J
			111% to 125%	Non-detect = No action, Detect = J
			<75%	Non-detect = UR, Detect = J
			>125%	Non-detect = No action Detect = J
Method blank	1/Preparation Batch (20 Samples)	No target analytes > 1/2 LOQ	Detected sample results <LOQ and <blank action level (BAL)	U at the LOQ



**QAPP Worksheet #36G-A Validation Data Qualification Summary**

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Method blank (continued)			Detected sample results >LOQ and <blank action level (BAL)	U at detected sample concentration
Interference Check Standards (ICS - ICSA & ICSB)	At the beginning of an analytical run.	ICSA recoveries must be within 80-120% of the true value. Analytes not present in ICSA must be less than 2x the absolute value of the limit of detection (LOD). ICSAB recoveries must be within 80-120 %R of the true value.	ICS %R>120	Detect = J
			ICS%R 50%-79%	Non-detect =UJ Detect = J
			ICSAB %R<50%	Non-detect = UR, Detect = R
			For samples containing comparable or higher levels of interferents and containing positive results > 2x LOD for analytes not present in the ICSA  For samples containing comparable or higher levels of interferents and containing results > 2x the absolute value of the LOD for analytes not present in the ICSA	Non-detect = UJ Detect = J
Laboratory Control Sample	1/Preparation Batch (20 Samples))	All aqueous LCS results should fall within lab control limits	LCS sample result > upper limit	Detect = J
			Lower limit - 5%< LCS sample result < lower limit	Non-detect = UJ, Detect = J
			LCS sample result < lower limit - 5%	Non-detect = UR, Detect = J



## QAPP Worksheet #36G-A Validation Data Qualification Summary

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Matrix Spike/Matrix Spike Duplicate	1/Preparation Batch (20 Samples)	80-120% Recovery RPD < 20% between MS and MSD for sample results < 4x the spike added	MS/MSD sample percent recovery 30-79%	Non-detect = UJ, Detect = J
			MS/MSD sample percent recovery < 30%	Non-detect = UR, Detect = J
			MS/MSD sample percent recovery >120% and/or RPD>20%	Non-detect = No action, Detect = J
Post Digestion Spike	When dilution test fails for analytes >100x MDL or when matrix spike recoveries fall outside of criteria an analyte concentration is <4x the spike added concentration.	%R 75% to 125%	%R>125%	Detect = J
			%R≥ 30 and ≤ 75%	Non-detect = UJ, Detect = J
			%R < 30%	Detect = J Non-detect = UR
Serial dilution	One per preparatory batch.	Five-fold dilution must agree within ±10% of the original measurement.	Sample concentration >50 times MDL and %D>10%	Non-detect =UJ Detect = J
Internal standards	Every sample	Internal standard intensity 30-120% of intensity of the internal standard in the initial calibration.	If less than or greater than the 30-120% range	Non-detect =UJ Detect = J
			No internal standards	Non-detect =UR Detect = R



**QAPP Worksheet #36G Validation (Steps IIa and IIb) Summary**

Step IIa/IIb	Matrix	Analytical Group	Data Purpose	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
Steps IIa and IIb	Groundwater	Mercury	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA <i>Contract Laboratory Program National Functional Guidelines for Inorganic Data Review</i> , EPA 540-R-04-004, October 2004; SW-846 method-specific criteria, DOD QSM, laboratory SOPs and control limits, QAPP Worksheet 12 and 28, and professional judgment.	Tetra Tech

During the data review process, laboratory data are evaluated against the requirements outlined Worksheet #36. Based on this evaluation, qualifiers may be added, removed, or modified by the data reviewer. Results are flagged with the following qualifiers in accordance with the USEPA National Functional Guidelines:

- Validation Qualifiers

- J The analyte was positively identified; however, the associated numerical value is an estimated concentration only.
- UJ The analyte was not detected above the reported sample detection limit. However, the reported limit is approximate and may or may not represent the actual limit of detection (LOD).
- U The analyte was analyzed for but not detected. The associated value is the analyte limit of detection.
- UR Nondetected sample results are rejected.
- R Positive sample results are rejected.

The "R" and "UR" flags mean that the associated result is unusable. In other words, due to significant quality control problems, the analysis is invalid and provides no usable information as to whether the analyte is present or not. "R" and "UR" results should not appear on data tables because these data points are not considered reliable.



QAPP Worksheet #36G-A Validation Data Qualification Summary				
Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Holding Time	Each environmental sample	28 days from collection to analysis	> 28 days to analysis	Non-detect = UR Detect = J
Sample preservation	Each environmental sample	$\leq 6^{\circ}\text{C}$	$> 6^{\circ}\text{C}$	Non-detect = UR Detect = J
Initial Calibration - Minimum of a five point calibration for all target analytes.	Initial Calibration performed daily prior to sample analysis.	Correlation Coefficient: $r \geq 0.995$ .	Correlation Coefficient: $r \geq 0.995$	Non-detect = UJ, Detect = J
Second-source calibration verification and Continuing calibration verification (CCV)	Second source - Immediately after calibration curve.  CCV – prior to sample analysis, after every 10 samples, and at the end of an analytical sequence.	All analytes within +/- 10% of true value	75% to 89%	Non-detect = UJ, Detect = J
			111% to 125%	Non-detect = No action, Detect = J
			<75%	Non-detect = UR, Detect = J
			>125%	Non-detect = No action Detect = J
Initial calibration blank and Continuing calibration blank (ICB)/(CCB)	Immediately after calibration curve and prior to sample analysis, after every 10 samples, and at the end of analytical sequence.	No analytes detected >1/2 Limit of Quantitation (LOQ)	Detected sample results <LOQ and <blank action level (BAL)	U at the LOQ



**QAPP Worksheet #36G-A Validation Data Qualification Summary**

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
(ICB)/(CCB) (Continued)			Detected sample results >LOQ and <blank action level (BAL)	U at detected sample concentration
Method blank	1/Preparation Batch (20 Samples)	No target analytes > ½ LOQ	Detected sample results <LOQ and <blank action level (BAL)	U at the LOQ
			Detected sample results >LOQ and <blank action level (BAL)	U at detected sample concentration
Laboratory Control Sample	1/Preparation Batch (20 Samples)	All aqueous LCS results should fall within lab control limits.	LCS sample result > upper limit	Non-detect = No action, Detect = J
			lower limit - 5% < LCS sample result < lower limit	Non-detect = UJ, Detect = J
			LCS sample result < lower limit - 5%	Non-detect = UR, Detect = J
Matrix Spike/Matrix Spike Duplicate	1/Preparation Batch (20 Samples)	80%-120% Recovery RPD < 20% between MS and MSD	MS/MDS sample percent recovery 30-79%	Non-detect = UJ, Detect = J
			MS/MSD sample percent recovery < 30%	Non-detect = UR, Detect = J
			MS/MSD sample percent recovery >120% and/or RPD>20%	Non-detect = No action, Detect = J



**QAPP Worksheet #36G Validation (Steps IIa and IIb) Summary**

Step IIa/IIb	Matrix	Analytical Group	Data Purpose	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
Steps IIa and IIb	Groundwater	PAHs	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA <i>Contract Laboratory Program National Functional Guidelines for Organic Data Review</i> , October 1999 (EPA540/R-99/008), SW-846 method-specific criteria, DOD QSM, laboratory SOPs and control limits, QAPP Worksheet 12 and 28, and professional judgment.	Tetra Tech

- Validation Qualifiers

- J The analyte was positively identified; however, the associated numerical value is an estimated concentration only.
- UJ The analyte was not detected above the reported sample detection limit. However, the reported limit is approximate and may or may not represent the actual limit of detection (LOD).
- U The analyte was analyzed for but not detected. The associated value is the analyte limit of detection (LOD).
- UR Nondetected sample results are rejected.
- R Positive sample results are rejected.

The "R" and "UR" flags mean that the associated result is unusable. In other words, due to significant quality control problems, the analysis is invalid and provides no usable information as to whether the analyte is present or not. "R" and "UR" results should not appear on data tables because these data points are not considered reliable.



## QAPP Worksheet #36G-A Validation Data Qualification Summary

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Holding Time	Each environmental sample	Holding time - 7 days from collection to extraction; 40 days from extraction to analysis.	>7 days to extraction	Non-detect = UR Detect = J
			>40 days to analysis	Non-detect = UR Detect = J
Sample preservation	Each environmental sample	0 to 6 °C	>6 °C	Non-detect = UR Detect = J
Tune	Before each initial calibration and every 12 hours in which samples or standards are analyzed.	Method specific ion abundance criteria must be met.	Relative ion abundances must be within specified limits.	Professional judgement
Initial Calibration - Minimum of a five point calibration for all target analytes.	Initial Calibration after initial setup and prior to sample analysis, and when continuing calibration verification criteria not met.	Calibration Check Compounds (CCC) percent Relative Standard Deviation (RSD) < 30% Other Analytes: Initial RSD ≤ 15% or $r^2 \geq 0.990$ Relative Response Factor (RRF) ≥ 0.05	CCC RSD < 30%; SPCC ≥ 0.05; Other Analytes: Initial RSD ≤ 15% or $r^2 \geq 0.990$	Non-detect = UJ, Detect = J
Second-source calibration verification	Once after each initial calibration.	+20% of true value for each individual compound	Compound percent recovery 80 - 120%	Data are not qualified based upon second source calibration verification noncompliance. The lab must correct noncompliance.



## QAPP Worksheet #36G-A Validation Data Qualification Summary

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Continuing calibration verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after decafluorotriphenylphosphine (DFTPP) tune.	The minimum Relative Response Factor (RRF) must be $\geq 0.05$ The CCCs must be $< 20$ percent difference.	Compound percent difference $> 20\%$	Non-detect = UJ, Detect = J
			Compound RRF $< 0.050$	Non-detect = R, Detect = J
Method blank	1/Preparation Batch (20 Samples)	No target analytes $> \frac{1}{2}$ LOQ	Detected sample results $< \text{LOQ}$ and $< \text{BAL}$	U at the LOQ
			Detected sample results $> \text{LOQ}$ and $< \text{BAL}$	U at detected sample concentration
Surrogate	3 base neutral fraction compounds are added to each sample and quality control sample prior to extraction.	Nitrobenzene-d5 %R= 40-110% 2-Fluorobiphenyl %R= 50-110% Terphenyl-d14 %R= 50-135%	Percent recovery 10% to lower limit (LL).	Non-detect = UJ, Detect = J
			Percent recovery $< 10\%$	Non-detect = UR, Detect = J
			Percent recovery $>$ upper limit (UL)	Non-detect = No action, Detect = J
Laboratory Control Sample	1/Preparation Batch (20 Samples))	Table G-6 (aqueous) QSM Version 4.1 (see attached Appendix A)	lower limit - $5\% < \%R <$ lower quality control limit.	Non-detect = UJ, Detect = J
			$\%R <$ lower limit - $5\%$	Non-detect = UR, Detect = J
			Percent Recovery greater than upper quality control limit.  RPD $> 30\%$	Non-detect = No action, Detect = J



## QAPP Worksheet #36G-A Validation Data Qualification Summary

Quality Control Requirements	Frequency	Quality Control Acceptance Criteria	Evaluation Limit	Data Qualification
Matrix Spike/Matrix Spike Duplicate	1/Preparation Batch (20 Samples)	Table G-6 (aqueous) QSM Version 4.1 (see attached Appendix A)	lower limit - 5% < %R < lower quality control limit.	Non-detect = UJ, Detect = J
			%R < lower limit - 5%	Non-detect = UR, Detect = J
			%R > than upper quality control limit.	Non-detect = No action Detect = J
			RPD > 30%	
Internal Standard	Six standards are added to all samples and quality control samples and standards	Retention time $\pm 30$ seconds from retention time of the midpoint standard in the initial calibration.  Area within -50% to +100% of the initial calibration midpoint standard.	> $\pm 30$ seconds	Professional judgement
			Percent recovery > 200%	Detect = J
			Percent recovery < 50%	Non-detect = UJ Detect = J
			Percent recovery < 25%	Non-detect = UR Detect = J



## **Attachment 1**

Chain of Custody





TETRA TECH NUS, INC.

CHAIN OF CUSTODY

NUMBER 3413

PAGE \_\_\_\_ OF \_\_\_\_

PROJECT NO:		FACILITY:		PROJECT MANAGER		PHONE NUMBER		LABORATORY NAME AND CONTACT:		
SAMPLERS (SIGNATURE)				FIELD OPERATIONS LEADER		PHONE NUMBER		ADDRESS		
				CARRIER/WAYBILL NUMBER		CITY, STATE				
STANDARD TAT <input type="checkbox"/> RUSH TAT <input type="checkbox"/> <input type="checkbox"/> 24 hr. <input type="checkbox"/> 48 hr. <input type="checkbox"/> 72 hr. <input type="checkbox"/> 7 day <input type="checkbox"/> 14 day				TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G)	
									PRESERVATIVE USED	
DATE YEAR	TIME	SAMPLE ID	LOCATION ID						TYPE OF ANALYSIS	
1. RELINQUISHED BY				DATE	TIME	1. RECEIVED BY			DATE	TIME
2. RELINQUISHED BY				DATE	TIME	2. RECEIVED BY			DATE	TIME
3. RELINQUISHED BY				DATE	TIME	3. RECEIVED BY			DATE	TIME
COMMENTS										

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE)

YELLOW (FIELD COPY)

PINK (FILE COPY)

4/02R  
FORM NO. TINUS-001

CHAIN-OF-CUSTODY RECORD FORM



## **Attachment 2**

Laboratory Certification





SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TRIMATRIX LABORATORIES INC.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512  
Rick Wilburn Phone: 616 975 4500

ENVIRONMENTAL

Valid To: April 30, 2011

Certificate Number: 0675.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1)) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a) as it pertains to the A2LA Kentucky Underground Storage Tank Laboratory Accreditation Program:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, Cold Vapor Atomic Fluorescence Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, F<sup>-</sup>, O<sub>2</sub>), Hazardous Waste Characteristic Tests, Spectrophotometry (Visible), Spectrophotometry (Automated) and Titrimetry.

<u>Parameter/Analyte</u>	<u>Potable Water</u> <u>(Non DoD)</u>	<u>Nonpotable Water</u> <u>(Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous</u> <u>(Non DoD and DoD)</u>	<u>Solid</u> <u>(Non DoD)</u>	<u>Solid</u> <u>(DoD)</u>
<b><u>Metals</u></b>					
Aluminum	EPA 200.7	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Antimony	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Arsenic	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Barium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Beryllium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Boron	-----	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Cadmium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A

*Peter M. Meyer*



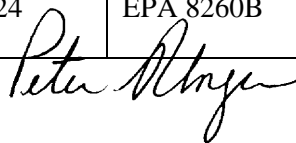
<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Calcium	EPA 200.7	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Chromium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Cobalt	-----	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Copper	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Iron	EPA 200.7	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Lead	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Lithium	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Magnesium	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Manganese	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Mercury	EPA 245.1	EPA 245.1 EPA 1631E	EPA 7470A	EPA 7471A	EPA 7471A
Molybdenum	-----	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Nickel	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Potassium	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Selenium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Silica (SiO <sub>2</sub> )	-----	SM 4500- SiO <sub>2</sub> D EPA 200.7	EPA 6010C	-----	-----
Silicon (Dissolved)	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Silver	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Sodium	EPA 200.7	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Strontium	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Thallium	EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Tin	-----	EPA 200.7 Tri GR-01- 129 ICPMS	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Titanium	-----	EPA 200.7	EPA 6010C	EPA 6010C	EPA 6010C
Vanadium	-----	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Zinc	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
<b><u>Nutrients</u></b>					
Ammonia (as N)	-----	SM 4500- NH <sub>3</sub> G	SM 4500- NH <sub>3</sub> G	SM 4500- NH <sub>3</sub> G	SM 4500- NH <sub>3</sub> G



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Nitrate (as N)	EPA 300.0 SM 4500- NO <sub>3</sub> F	EPA 300.0 SM 4500- NO <sub>3</sub> F	EPA 9056A SM 4500- NO <sub>3</sub> F	EPA 9056A	EPA 9056A
Nitrate-nitrite (as N)	EPA 300.0 SM 4500- NO <sub>3</sub> F	EPA 300.0 SM 4500- NO <sub>3</sub> F	EPA 9056A SM 4500- NO <sub>3</sub> F	EPA 9056A	EPA 9056A
Nitrite (as N)	EPA 300.0 SM 4500- NO <sub>2</sub> B SM 4500- NO <sub>3</sub> F	EPA 300.0 SM 4500- NO <sub>2</sub> B SM 4500- NO <sub>3</sub> F	EPA 9056A SM 4500- NO <sub>2</sub> B SM 4500- NO <sub>3</sub> F	EPA 9056A	EPA 9056A
Orthophosphate (as P)	SM 4500-P E	SM 4500-P E	SM 4500-P E	-----	-----
Total phosphorus	-----	SM 4500-P E	SM 4500-P E	SM 4500-P E	SM 4500-P E
Total kjeldahl nitrogen	-----	EPA 351.2	EPA 351.2	-----	-----
<b><u>Demands</u></b>					
Biochemical oxygen demand	-----	SM 5210 B	-----	-----	-----
Carbonaceous BOD	-----	SM 5210 B	-----	-----	-----
Chemical oxygen demand	-----	SM 5220 D	-----	-----	-----
Total organic carbon	SM 5310 C	SM 5310 C	EPA 9060	Walkley Black (MSA 29-3.5-2)	Walkley Black (MSA 29-3.5-2)
<b><u>Wet Chemistry</u></b>					
Acidity	-----	SM 2310 B	-----	-----	-----
Alkalinity	SM 2320 B	SM 2320 B	-----	-----	-----
Bromide	-----	EPA 300.0	EPA 9056A	EPA 9056A	EPA 9056A
Chloride	EPA 300.0	EPA 300.0 SM 4500- Cl E	EPA 9056A SM 4500- Cl E	EPA 9056A	EPA 9056A
Chromium (VI) Cr 6+	-----	SM 3500-Cr B	EPA 7196A	EPA 7196A	EPA 7196A
Cyanide	SM 4500- CN E	SM 4500- CN E	EPA 9014	EPA 9014	EPA 9014
Cyanide Amenable to Chlorination	-----	SM 4500- CN G	EPA 9014	EPA 9014	EPA 9014
Fluoride	SM 4500-F C	EPA 300.0 SM 4500- F C	EPA 9056A SM 4500- F C	EPA 9056A SM 4500- F C	EPA 9056A SM 4500-F C
Hardness (total and calcium)	EPA 200.7 SM 2340 B SM 2340 C	SM 2340 B SM 2340 C	-----	-----	-----
Oil and Grease / TPH	-----	EPA 1664A	EPA 1664A EPA 9070A	EPA 9071B	EPA 9071B
pH	SM 4500-H B	SM 4500-H B	EPA 9040B	EPA 9045C	EPA 9045C
Phenolics	-----	EPA 420.4	EPA 9065	EPA 9065	EPA 9065
Conductivity	SM 2510 B	SM 2510 B	EPA 9050A	-----	-----
Sulfate	EPA 300.0	EPA 300.0 ASTM D 516-90 (02)	EPA 9038 EPA 9056A	EPA 9038 EPA 9056A	EPA 9038 EPA 9056A



<b><u>Parameter/Analyte</u></b>	<b><u>Potable Water (Non DoD)</u></b>	<b><u>Nonpotable Water (Non DoD and DoD)</u></b>	<b><u>Solid Hazardous Waste</u></b>		
			<b><u>Aqueous (Non DoD and DoD)</u></b>	<b><u>Solid (Non DoD)</u></b>	<b><u>Solid (DoD)</u></b>
Sulfide	-----	SM 4500-S <sub>2</sub> D, F	EPA 9030B EPA 9034	EPA 9030B EPA 9034	EPA 9030B EPA 9034
Sulfite	-----	SM 4500-SO <sub>3</sub> B	-----	-----	-----
Surfactants (MBAS)	-----	SM 5540 C	-----	-----	-----
Total Residue	-----	SM 2540 B	-----	Tri GR-07-115 Grav	Tri GR-07-115 Grav
Dissolved Residue (TDS)	SM 2540 C	SM 2540 C	-----	-----	-----
Suspended Residue (TSS)	-----	SM 2540 D	-----	-----	-----
Turbidity	SM 2130 B	SM 2130 B	-----	-----	-----
Volatile Residue	-----	EPA 160.4	-----	-----	-----
Color	EPA 2120 B	EPA 2120 B	-----	-----	-----
Total Residual Chlorine	Hach 8167	Hach 8167	-----	-----	-----
Available Cyanide	-----	OIA-1677	OIA-1677	-----	-----
<b><u>Purgeable Organics</u></b>					
Acetone	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Acetonitrile	-----	EPA 624	EPA 8260B	EPA 8260B	EPA 8260B
Acrolein	-----	EPA 624	EPA 8260B	EPA 8260B	EPA 8260B
Acrylonitrile	-----	EPA 624	EPA 8260B	EPA 8260B	EPA 8260B
Allyl Chloride (3-chloropropene)	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Benzene	EPA 524.2	EPA 602 EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA 8021B	EPA 8260B
Bromobenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Bromochloromethane	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Bromodichloromethane	EPA 524.2	EPA 601 EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA 8021B	EPA 8260B
Bromoform	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Bromomethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Carbon disulfide	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Carbon tetrachloride	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Chlorobenzene	EPA 524.2	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Chloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Chloroform	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Chloromethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B





<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Chloroprene	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
1-Chlorohexane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Cyclohexane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
cis-1,2-Dichloroethylene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Dibromochloromethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Dibromomethane	EPA 524.2	-----	EPA 8260B	EPA 8260B	EPA 8260B
Dichlorodifluoromethane	EPA 524.2	EPA 601	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Diethyl ether	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Ethanol	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Ethyl Acetate	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Ethyl methacrylate	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Ethylbenzene	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Ethyl tert-Butyl Ether (EtBE)	-----	-----	EPA 8260B	EPA 8260B	-----
Gasoline Range Organics	-----	-----	EPA 8015B	EPA 8015B	EPA 8015B
Hexachlorobutadiene	EPA 524.2	-----	EPA 8260B	EPA 8260B	EPA 8260B
Hexachloroethane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Iodomethane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Isobutyl alcohol	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Isopropyl alcohol (2-Propanol)	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Isopropyl ether	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Ethane	-----	RSK-175	-----	-----	-----
Ethylene	-----	RSK-175	-----	-----	-----
Methane	-----	RSK-175	-----	-----	-----
Isopropylbenzene	EPA 524.2	-----	EPA 8260B	EPA 8260B	EPA 8260B
Methacrylonitrile	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Methanol	-----	-----	EPA 8015B	EPA 8015B	EPA 8015B
Methylcyclohexane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Methylene chloride	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Methyl ethyl ketone (MEK) (2-Butanone)	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Methyl methacrylate	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Methyl tert-butyl ether (MTBE)	-----	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
2-Nitropropane	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Naphthalene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
n-Butyl alcohol	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
n-Butylbenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
n-Propyl alcohol	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
n-Propylbenzene	EPA 524.2	-----	EPA 8260B	EPA 8260B	EPA 8260B
Paraldehyde	-----	-----	EPA 8015B	EPA 8015B	EPA 8015B
Propionitrile	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Sec-Butylbenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Styrene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Freon-113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	-----	EPA 624	EPA 8260B	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1-Dichloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1-Dichloroethene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,1-Dichloropropene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2,4-Trimethylbenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dibromo-3-chloropropane (DBCP)	-----	-----	EPA 8021B EPA 8260B EPA 8011	EPA 8021B EPA 8260B	EPA 8260B

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<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
1,2-Dibromoethane (EDB)	-----	-----	EPA 8021B EPA 8260B EPA 8011	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dichlorobenzene	EPA 524.2	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dichloroethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dichloropropane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,3-Dichlorobenzene	EPA 524.2	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,3-Dichloropropane	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,4-Dichlorobenzene	EPA 524.2	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
1,4-Dioxane (p-Dioxane)	-----	-----	EPA 8021B EPA 8260B EPA 8270C	EPA 8270C EPA 8021B EPA 8260B	EPA 8260B
2,2-Dichloropropane	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	-----	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
2-Chlorotoluene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
2-Hexanone	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
4-Chlorotoluene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
4-Isopropyltoluene	EPA 524.2	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
4-Methyl-2-pentanone (MIBK)	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
tert-Amyl methyl ether (TAME)	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
tert-Butyl alcohol	-----	-----	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B	EPA 8260B
tert-Butylbenzene	-----	-----	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Tetrachloroethene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Toluene	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B

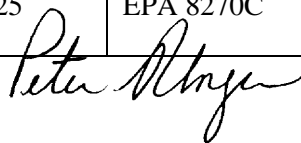
*Peter M. Meyer*



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
trans-1,2-Dichloroethene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Trichloroethylene	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Trichlorofluoromethane	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Trihalomethanes (Total)	EPA 524.2	-----	-----	-----	-----
Vinyl acetate	-----	-----	EPA 8260B	EPA 8260B	EPA 8260B
Vinyl chloride	EPA 524.2	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
m-Xylene	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
o-Xylene	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
p-Xylene	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
Xylenes Total	EPA 524.2	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8021B EPA 8260B	EPA 8260B
<b><u>Extractable Organics (Semivolatiles)</u></b>					
Acenaphthene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Acenaphthylene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Acetophenone	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Acetaldehyde	-----	-----	EPA 8315A	EPA 8315A	-----
2-Acetylaminofluorene	-----	-----	EPA 8270C	EPA 8270C	-----
4-Aminobiphenyl	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
2-Amino-4,6-dinitrotoluene	-----	-----	EPA 8330	EPA 8330	EPA 8330
4-Amino-2,6-dinitrotoluene	-----	-----	EPA 8330	EPA 8330	EPA 8330
Aniline	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Anthracene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Aramite	-----	-----	EPA 8270C	EPA 8270C	-----
Benzidine	-----	EPA 625	EPA 8270C	EPA 8270C	-----
Benzoic acid	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Benzaldehyde	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Benzo(a)anthracene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(b)fluoranthene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(k)fluoranthene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Benzo(ghi)perylene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(a)pyrene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzyl alcohol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Biphenyl	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Bis (2-Chloroethoxy) methane	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Bis (2-Chloroethyl) ether	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Bis (2-Chloroisopropyl) ether	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Bis (2-Ethylhexyl) phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
4-Bromophenyl phenyl ether	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Butanal	-----	-----	EPA 8315A	EPA 8315A	-----
Butyl benzyl phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Caprolactam	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Carbazole	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
4-Chloroaniline	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2-Chloronaphthalene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2-Chlorophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Chrysene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
4-Chloro-3-methylphenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
4-Chlorophenyl phenyl ether	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Crotonaldehyde	-----	-----	EPA 8315A	EPA 8315A	-----
Cyclohexanone	-----	-----	EPA 8315A	EPA 8315A	-----
Decanal	-----	-----	EPA 8315A	EPA 8315A	-----
Dibenzo(a,h)anthracene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Dibenzofuran	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
1,2-Dichlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
1,3-Dichlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
1,4-Dichlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
3,3-Dichlorobenzidine	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2,4-Dichlorophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2,6-Dichlorophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Diethyl phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Diesel Range Organics (DRO)	-----	-----	EPA 8015B	EPA 8015B	EPA 8015B
Dimethoate	-----	-----	EPA 8270C	EPA 8270C	-----
p-Dimethylaminoazobenzene	-----	-----	EPA 8270C	EPA 8270C	-----
7,12-Dimethylbenz(a)anthracene	-----	-----	EPA 8270C	EPA 8270C	-----
3,3'-Dimethylbenzidine	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Alpha-alpha-Dimethylphenethylamine	-----	-----	EPA 8270C	EPA 8270C	-----
2,4-Dimethylphenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C





<b>Parameter/Analyte</b>	<b>Potable Water (Non DoD)</b>	<b>Nonpotable Water (Non DoD and DoD)</b>	<b>Solid Hazardous Waste</b>		
			<b>Aqueous (Non DoD and DoD)</b>	<b>Solid (Non DoD)</b>	<b>Solid (DoD)</b>
Dimethyl phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Di-n-butyl phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Di-n-octyl phthalate	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Dinoseb (2-sec-butyl-4,6-dinitrophenol)	-----	-----	EPA 8270C EPA 8151A	EPA 8270C EPA 8151A	EPA 8151A
2,4-Dinitrophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2,4-Dinitrotoluene	-----	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330	EPA 8270C EPA 8330
2,6-Dinitrotoluene	-----	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330	EPA 8270C EPA 8330
1,3-Dinitrobenzene	-----	-----	EPA 8270C EPA 8330	EPA 8270C EPA 8330	EPA 8270C EPA 8330
1,2-Diphenylhydrazine	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Diphenylamine	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Disulfoton	-----	-----	EPA 8270C	EPA 8270C	-----
Ethyl methanesulfonate	-----	-----	EPA 8270C	EPA 8270C	-----
Famphur	-----	-----	EPA 8270C	EPA 8270C	-----
Fluoranthene	-----	EPA 625 EPA 610	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Fluorene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Formaldehyde	-----	-----	EPA 8315A	EPA 8315A	-----
Heptanal	-----	-----	EPA 8315A	EPA 8315A	-----
Hexachlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Hexachlorobutadiene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Hexachlorocyclopentadiene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Hexachloroethane	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Hexachloropropene	-----	-----	EPA 8270C	EPA 8270C	-----
Hexanal	-----	-----	EPA 8315A	EPA 8315A	-----
HMX	-----	-----	EPA 8330	EPA 8330	EPA 8330
Indeno(1,2,3-cd)pyrene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Isophorone	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Isosafrole	-----	-----	EPA 8270C	EPA 8270C	-----
Kepone	-----	-----	EPA 8270C EPA 8081A	EPA 8270C EPA 8081A	EPA 8270C
Methapyrilene	-----	-----	EPA 8270C	EPA 8270C	-----
3-Methylcholanthrene	-----	-----	EPA 8270C	EPA 8270C	-----
2-Methyl-4,6-dinitrophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Methyl methanesulfonate	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
1-Methylnaphthalene	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
2-Methylnaphthalene	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
2-Methylphenol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
3-Methylphenol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
4-Methylphenol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Naphthalene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
1,4-Naphthoquinone	-----	-----	EPA 8270C	EPA 8270C	-----
1-Naphthylamine	-----	-----	EPA 8270C	EPA 8270C	-----
2-Naphthylamine	-----	-----	EPA 8270C	EPA 8270C	-----
2-Nitroaniline	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
3-Nitroaniline	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
4-Nitroaniline	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Nitrobenzene	-----	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330	EPA 8270C EPA 8330
Nitroquinoline-1-oxide	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
2-Nitrophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
4-Nitrophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
n-Nitrosodiethylamine	-----	-----	EPA 8270C	EPA 8270C	-----
n-Nitroso-di-n-butylamine	-----	-----	EPA 8270C	EPA 8270C	-----
n-Nitroso-di-methylamine	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
n-Nitroso-di-n-propylamine	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
n-Nitroso-di-phenylamine	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
n-Nitrosomethylethylamine	-----	-----	EPA 8270C	EPA 8270C	-----
n-Nitrosomorpholine	-----	-----	EPA 8270C	EPA 8270C	-----
n-Nitrosopiperidine	-----	-----	EPA 8270C	EPA 8270C	-----
n-Nitrosopyrrolidine	-----	-----	EPA 8270C	EPA 8270C	-----
2-Nitrotoluene	-----	-----	EPA 8330	EPA 8330	EPA 8330
3-Nitrotoluene	-----	-----	EPA 8330	EPA 8330	EPA 8330
4-Nitrotoluene	-----	-----	EPA 8330	EPA 8330	EPA 8330
5-Nitro-o-toluidine	-----	-----	EPA 8270C	EPA 8270C	-----
Nonanal	-----	-----	EPA 8315A	EPA 8315A	-----
Octanal	-----	-----	EPA 8315A	EPA 8315A	-----
O,O,O-Triethyl phosphorothioate	-----	-----	EPA 8270C	EPA 8270C	-----
o-Toluidine	-----	-----	EPA 8270C	EPA 8270C	-----
Ethyl parathion	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Methyl parathion	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
Pentachlorobenzene	-----	-----	EPA 8270C	EPA 8270C	-----
Pentachloronitrobenzene	-----	-----	EPA 8270C	EPA 8270C	-----
Pentachlorophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Pentanal	-----	-----	EPA 8315A	EPA 8315A	-----
Phenacetin	-----	-----	EPA 8270C	EPA 8270C	-----
Phenanthrene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Phenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
1,4-Phenylenediamine	-----	-----	EPA 8270C	EPA 8270C	-----
Phorate	-----	-----	EPA 8270C	EPA 8270C	-----
2-Picoline	-----	-----	EPA 8270C	EPA 8270C	-----

*Peter M. Meyer*



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Pronamide	-----	-----	EPA 8270C	EPA 8270C	-----
Propanal (Propionaldehyde)	-----	-----	EPA 8315A	EPA 8315A	-----
Pyrene	-----	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Pyridine	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
RDX	-----	-----	EPA 8330	EPA 8330	EPA 8330
Safrole	-----	-----	EPA 8270C	EPA 8270C	-----
Sulfotepp	-----	-----	EPA 8270C	EPA 8270C	-----
Tetryl	-----	-----	EPA 8330	EPA 8330	EPA 8330
1,2,4,5-Tetrachlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Thionazin	-----	-----	EPA 8270C	EPA 8270C	-----
1,3-Tolualdehyde	-----	-----	EPA 8315A	EPA 8315A	-----
1,2,4-Trichlorobenzene	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
2,3,4,6-Tetrachlorophenol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
1,3,5-Trinitrobenzene	-----	-----	EPA 8330	EPA 8330	EPA 8330
2,4,6-Trinitrotoluene	-----	-----	EPA 8270C EPA 8330	EPA 8270C EPA 8330	EPA 8330
2,4,5-Trichlorophenol	-----	-----	EPA 8270C	EPA 8270C	EPA 8270C
2,4,6-Trichlorophenol	-----	EPA 625	EPA 8270C	EPA 8270C	EPA 8270C
Nitroglycerine	-----	-----	EPA 8332	EPA 8332	EPA 8332
PETN	-----	-----	EPA 8332	EPA 8332	EPA 8332
<b><u>Pesticides-Herbicides-PCBs</u></b>					
Aldrin	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
a-BHC	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
b-BHC	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
d-BHC	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
g-BHC (Lindane)	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A	EPA 8081A
Chlorobenzilate	-----	-----	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8270C
Chlordane	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8081A
2,4-D	-----	-----	EPA 8151A EPA 8270C	EPA 8151A EPA 8270C	EPA 8151A
Dalapon	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
2,4,5-T	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Silvex (2,4,5-TP)	-----	-----	EPA 8151A EPA 8270C	EPA 8151A	EPA 8151A
4,4'-DDD	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
4,4' DDE	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
4,4'-DDT	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Diallate	-----	-----	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8270C



<u>Parameter/Analyte</u>	<u>Potable Water (Non DoD)</u>	<u>Nonpotable Water (Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous (Non DoD and DoD)</u>	<u>Solid (Non DoD)</u>	<u>Solid (DoD)</u>
Dicamba	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Dichloroprop	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Dieldrin	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Dinoseb	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Endosulfan I (alpha)	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Endosulfan II (beta)	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Endosulfan sulfate	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Endrin	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8081A
Endrin aldehyde	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Endrin ketone	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
a-Chlordane	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
g-Chlordane	-----	EPA 608	EPA 8081A	EPA 8081A	EPA 8081A
Heptachlor	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8081A
Heptachlor epoxide	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A	EPA 8081A
Isodrin	-----	-----	EPA 8081A EPA 8270C	EPA 8081A EPA 8270C	EPA 8270C
Methoxychlor	-----	EPA 608.2	EPA 8081A EPA 8270C	EPA 8081A	EPA 8081A
MCPA	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Toxaphene	-----	EPA 608	EPA 8081A EPA 8270C	EPA 8081A	EPA 8081A
Picloram	-----	-----	EPA 8151A	EPA 8151A	-----
PCB 1016	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1221	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1232	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1242	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1248	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1254	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1260	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1262	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
PCB 1268	-----	EPA 608	EPA 8082	EPA 8082	EPA 8082
<b><u>Hazardous Waste Characteristics</u></b>					
Corrosivity	-----	-----	EPA 9040B	EPA 9045C	EPA 9045C
Ignitability	-----	-----	-----	EPA 1020A	EPA 1020A
Paint Filter Liquid Test	-----	-----	-----	EPA 9095A	EPA 9095A
TCLP	-----	-----	EPA 1311	EPA 1311	EPA 1311
SPLP	-----	-----	EPA 1312	EPA 1312	EPA 1312
Total Organic Halides	-----	-----	EPA 9020B	-----	-----



<u>Parameter/Analyte</u>	<u>Potable Water</u> <u>(Non DoD)</u>	<u>Nonpotable Water</u> <u>(Non DoD and DoD)</u>	<u>Solid Hazardous Waste</u>		
			<u>Aqueous</u> <u>(Non DoD and DoD)</u>	<u>Solid</u> <u>(Non DoD)</u>	<u>Solid</u> <u>(DoD)</u>
<b><u>Microbiology</u></b>					
Total Coliform	9223 B	-----	-----	-----	-----
Fecal Coliform	9222 D	-----	-----	-----	-----
Heterotrophic Plate Count	9215 B	-----	-----	-----	-----

**Field on- Site Sampling and Analysis\***

<u>Parameter/Analyte</u>	<u>Potable Water</u>	<u>Nonpotable Water</u>	<u>Solid Hazardous Waste</u> <u>Aqueous</u>	<u>Solid Hazardous Waste</u> <u>Solid</u>
pH	-----	SM 4500-H B	EPA 9040B	-----
Conductivity	-----	SM 2510 B	EPA 9050A	-----
Dissolved Oxygen	-----	SM 4500-O G	-----	-----
Temperature	-----	SM 2550 B	-----	-----
Redox	-----	ASTM D1498-76	-----	-----
Turbidity	-----	SM 2130 B	-----	-----
Sampling	-----	TRI GR-20-102 TRI GR-20-103 TRI GR-20-104 TRI GR-20-105 TRI GR-20-113 ASTM D6538	TRI GR-20-102 TRI GR-20-103 TRI GR-20-105 TRI GR-20-113 TRI GR-20-104	TRI GR-20-108

**Kentucky UST Program**

Gasoline Range Organics	-----	-----	EPA 8015B
Diesel Range Organics (DRO)	-----	-----	EPA 8015B
Oil and Grease / TPH	-----	-----	EPA 9071B
<b><u>TCLP Metals</u></b>			
Arsenic	-----	-----	EPA 6010C EPA 6020A
Barium	-----	-----	EPA 6010C EPA 6020A
Cadmium	-----	-----	EPA 6010C EPA 6020A
Chromium	-----	-----	EPA 6010C EPA 6020A
Lead	-----	-----	EPA 6010C EPA 6020A
Mercury	-----	-----	EPA 7471A
Selenium	-----	-----	EPA 6010C EPA 6020A
Silver	-----	-----	EPA 6010C EPA 6020A

*Peter Meyer*



**Preparation Methods**

<b>Fraction</b>	<b>Analytical Method</b>	<b>Preparation Method</b>
Metals by ICP	EPA 200.7 EPA 6010C	EPA 3005A EPA 3010A EPA 3020A EPA 3050B EPA 200.2
Metals by ICP/MS	EPA 200.8 EPA 6020A	EPA 3005A EPA 3010A EPA 3020A EPA 3050B EPA 200.2
Hexavalent Chromium	EPA 7196A	EPA 3060A
Organochlorine Pesticides	EPA 8081B	EPA 3510C EPA 3550B EPA 3545
Polychlorinated Biphenyls (PCBs)	EPA 8082	EPA 3510C EPA 3550B EPA 3545
BNAs	EPA 8270C	EPA 3510C EPA 3550B EPA 3545
Volatiles	EPA 8260B	EPA 5030B EPA 5035A
Diesel Range Organics	EPA 8015B	EPA 3510C EPA 3550B EPA 3545
Gasoline Range Organics	EPA 8015B	EPA 5030B EPA 5035A
Sulfide	EPA 9034	EPA 9030B
Cyanide	EPA 9014	EPA 9013A EPA 9010B

*\* Not accredited under DOD program requirement.*







State of Florida  
Department of Health, Bureau of Laboratories  
This is to certify that  
E87622

TRIMATRIX LABORATORIES, INC.  
5560 CORPORATE EXCHANGE COURT SE  
GRAND RAPIDS, MI 49512

has complied with Florida Administrative Code 64E-1,  
for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER -  
PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE  
ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER -  
PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS,  
SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS -  
PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

**EFFECTIVE July 01, 2010 THROUGH June 30, 2011**



A handwritten signature in black ink, appearing to read "Max Salfinger".

Max Salfinger, M.D.  
Chief, Bureau of Laboratories  
Florida Department of Health  
DH Form 1697, 7/04

NON-TRANSFERABLE E87622-13-07/01/2010  
Supersedes all previously issued certificates



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

Page 1 of 35

Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,2,4-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Alkalinity as CaCO <sub>3</sub>	SM 2320 B	Primary Inorganic Contaminants	NELAP	5/19/2003
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	1/24/2001
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Bromoform	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	1/24/2001
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

Page 2 of 35

Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.

5560 Corporate Exchange Court SE

Grand Rapids, MI 49512

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	5/19/2003
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Chloroform	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	7/24/2008
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	5/19/2003
Copper	EPA 200.7	Primary Inorganic Contaminants, Secondary Inorganic Contaminants	NELAP	1/24/2001
Copper	EPA 200.8	Primary Inorganic Contaminants, Secondary Inorganic Contaminants	NELAP	1/24/2001
Cyanide	SM 4500-CN E	Primary Inorganic Contaminants	NELAP	7/24/2008
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Fluoride	SM 4500 F-C	Primary Inorganic Contaminants	NELAP	5/19/2003
Hardness	SM 2340 B	Secondary Inorganic Contaminants	NELAP	7/24/2008
Hardness	SM 2340 C	Secondary Inorganic Contaminants	NELAP	7/24/2008
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	1/24/2001
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	1/24/2001
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	1/24/2001
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	5/19/2003
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Nitrate as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	5/19/2003
Nitrate as N	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	9/9/2005
Nitrite	SM 4500-NO2 B	Primary Inorganic Contaminants	NELAP	5/19/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011





*Laboratory Scope of Accreditation*

Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrite	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	9/9/2005
Nitrite as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	5/19/2003
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Orthophosphate as P	SM 4500-P E	Primary Inorganic Contaminants	NELAP	7/24/2008
pH	SM 4500-H B	Primary Inorganic Contaminants	NELAP	5/19/2003
Residue-filterable (TDS)	SM 2540 C	Secondary Inorganic Contaminants	NELAP	9/9/2005
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	1/24/2001
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	1/24/2001
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Sulfate	EPA 300.0	Secondary Inorganic Contaminants	NELAP	5/19/2003
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	1/24/2001
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	5/19/2003
Total nitrate-nitrite	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	9/9/2005
Total organic carbon	SM 5310 C	Primary Inorganic Contaminants	NELAP	7/24/2008
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	7/24/2008
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/19/2003
Turbidity	SM 2130 B	Secondary Inorganic Contaminants	NELAP	5/19/2003
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	5/19/2003
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	4/20/2001
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/20/2001





# *Laboratory Scope of Accreditation*

Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,1,1-Trichloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,1-Trichloroethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,1,2,2-Tetrachloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,2,2-Tetrachloroethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,1,2-Trichloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,2-Trichloroethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,1-Dichloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,1-Dichloroethylene	EPA 601	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,1-Dichloropropene	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2,3-Trichlorobenzene	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2,3-Trichloropropane	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/2/2005
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
1,2,4-Trichlorobenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8021	Volatile Organics	NELAP	7/24/2008

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,2-Dichlorobenzene	EPA 601	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 602	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,2-Dichloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2-Dichloropropane	EPA 601	Volatile Organics	NELAP	1/24/2001
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichloropropane	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/24/2008
1,3-Dichlorobenzene	EPA 601	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 602	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,3-Dichloropropane	EPA 8021	Volatile Organics	NELAP	7/24/2008
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/24/2008
1,3-Tolualdehyde	EPA 8315	Extractable Organics	NELAP	7/24/2008
1,4-Dichlorobenzene	EPA 601	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 602	Volatile Organics	NELAP	1/24/2001

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	9/2/2005
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1-Methylnaphthalene (added to method at FDEP request)	EPA 8270	Extractable Organics	NELAP	7/24/2008
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,2-Dichloropropane	EPA 8021	Volatile Organics	NELAP	7/24/2008
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/2/2005
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/24/2008
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	1/24/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	9/2/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/24/2008
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/24/2008
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	1/24/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	9/2/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/24/2008
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	9/2/2005
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/24/2008

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





*Laboratory Scope of Accreditation*

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/24/2008
2-Chloroethyl vinyl ether	EPA 601	Volatile Organics	NELAP	1/24/2001
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	1/24/2001
2-Chloroethyl vinyl ether	EPA 8021	Volatile Organics	NELAP	7/24/2008
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/24/2008
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	1/24/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	9/2/2005
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
2-Chlorotoluene	EPA 8021	Volatile Organics	NELAP	7/24/2008
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	9/2/2005
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/24/2008
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/24/2008
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/24/2008
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/24/2008
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/24/2008
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/24/2008
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/24/2008
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	9/2/2005
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	1/24/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	9/2/2005
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	9/2/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/24/2008
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/24/2008
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/24/2008
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	1/24/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	1/24/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/24/2008
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	1/24/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Chlorotoluene	EPA 8021	Volatile Organics	NELAP	7/24/2008
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	9/2/2005
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/24/2008
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/24/2008
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/24/2008
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/24/2008
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	9/2/2005
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
Acenaphthene	EPA 610	Extractable Organics	NELAP	7/24/2008
Acenaphthene	EPA 625	Extractable Organics	NELAP	1/24/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Acenaphthene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Acenaphthylene	EPA 610	Extractable Organics	NELAP	7/24/2008
Acenaphthylene	EPA 625	Extractable Organics	NELAP	1/24/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Acetaldehyde	EPA 8315	Extractable Organics	NELAP	7/24/2008
Acetone	EPA 8260	Volatile Organics	NELAP	7/24/2008
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/24/2008
Acetophenone	EPA 8270	Extractable Organics	NELAP	9/2/2005
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	5/19/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Acrylonitrile	EPA 624	Volatile Organics	NELAP	5/19/2003

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	9/2/2005
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Alkalinity as CaCO <sub>3</sub>	SM 2320 B	General Chemistry	NELAP	9/2/2005
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	9/2/2005
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Aluminum	EPA 200.7	Metals	NELAP	1/24/2001
Aluminum	EPA 6010	Metals	NELAP	9/2/2005
Amenable cyanide	SM 4500 CN-G	General Chemistry	NELAP	5/19/2003
Ammonia as N	SM 4500-NH <sub>3</sub> G	General Chemistry	NELAP	5/19/2003
Aniline	EPA 8270	Extractable Organics	NELAP	7/24/2008
Anthracene	EPA 610	Extractable Organics	NELAP	7/24/2008
Anthracene	EPA 625	Extractable Organics	NELAP	1/24/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Anthracene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Antimony	EPA 200.7	Metals	NELAP	1/24/2001
Antimony	EPA 200.8	Metals	NELAP	1/24/2001
Antimony	EPA 6010	Metals	NELAP	9/2/2005
Antimony	EPA 6020	Metals	NELAP	9/2/2005
Aramite	EPA 8270	Extractable Organics	NELAP	9/2/2005
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/2/2005

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





# *Laboratory Scope of Accreditation*

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Arsenic	EPA 200.7	Metals	NELAP	1/24/2001
Arsenic	EPA 200.8	Metals	NELAP	1/24/2001
Arsenic	EPA 6010	Metals	NELAP	9/2/2005
Arsenic	EPA 6020	Metals	NELAP	9/2/2005
Available cyanide	OIA 1677	General Chemistry	NELAP	9/9/2005
Barium	EPA 200.7	Metals	NELAP	1/24/2001
Barium	EPA 200.8	Metals	NELAP	1/24/2001
Barium	EPA 6010	Metals	NELAP	9/2/2005
Barium	EPA 6020	Metals	NELAP	9/2/2005
Benzene	EPA 602	Volatile Organics	NELAP	1/24/2001
Benzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Benzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
Benzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Benzidine	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(a)anthracene	EPA 610	Extractable Organics	NELAP	7/24/2008
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Benzo(a)pyrene	EPA 610	Extractable Organics	NELAP	7/24/2008
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Benzo(b)fluoranthene	EPA 610	Extractable Organics	NELAP	7/24/2008
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Benzo(g,h,i)perylene	EPA 610	Extractable Organics	NELAP	7/24/2008
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Benzo(k)fluoranthene	EPA 610	Extractable Organics	NELAP	7/24/2008
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	1/24/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/24/2008

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/24/2008
Beryllium	EPA 200.7	Metals	NELAP	1/24/2001
Beryllium	EPA 200.8	Metals	NELAP	1/24/2001
Beryllium	EPA 6010	Metals	NELAP	9/2/2005
Beryllium	EPA 6020	Metals	NELAP	9/2/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	5/19/2003
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	1/24/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	9/2/2005
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	1/24/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	9/2/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	7/24/2008
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/24/2008
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	1/24/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	9/2/2005
Boron	EPA 200.7	Metals	NELAP	1/24/2001
Boron	EPA 6010	Metals	NELAP	9/2/2005
Bromobenzene	EPA 8021	Volatile Organics	NELAP	7/24/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Bromochloromethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Bromodichloromethane	EPA 601	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Bromoform	EPA 601	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 624	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 8021	Volatile Organics	NELAP	9/2/2005
Bromoform	EPA 8260	Volatile Organics	NELAP	9/2/2005
Butanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	1/24/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Cadmium	EPA 200.7	Metals	NELAP	1/24/2001
Cadmium	EPA 200.8	Metals	NELAP	1/24/2001

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Cadmium	EPA 6010	Metals	NELAP	9/2/2005
Cadmium	EPA 6020	Metals	NELAP	9/2/2005
Calcium	EPA 200.7	Metals	NELAP	1/24/2001
Calcium	EPA 6010	Metals	NELAP	9/2/2005
Carbazole	EPA 8270	Extractable Organics	NELAP	7/24/2008
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/24/2008
Carbon tetrachloride	EPA 601	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 8021	Volatile Organics	NELAP	9/2/2005
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	9/2/2005
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	5/19/2003
Chemical oxygen demand	SM 5220 D	General Chemistry	NELAP	5/19/2003
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Chloride	EPA 300.0	General Chemistry	NELAP	5/19/2003
Chloride	EPA 9056	General Chemistry	NELAP	9/2/2005
Chloride	SM 4500-Cl E	General Chemistry	NELAP	5/19/2003
Chlorobenzene	EPA 601	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 602	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Chloroethane	EPA 601	Volatile Organics	NELAP	1/24/2001
Chloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Chloroethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
Chloroethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Chloroform	EPA 601	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 624	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 8021	Volatile Organics	NELAP	9/2/2005
Chloroform	EPA 8260	Volatile Organics	NELAP	9/2/2005
Chloroprene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Chromium	EPA 200.7	Metals	NELAP	1/24/2005
Chromium	EPA 200.8	Metals	NELAP	1/24/2001
Chromium	EPA 6010	Metals	NELAP	9/2/2005
Chromium	EPA 6020	Metals	NELAP	9/2/2005

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium VI	EPA 7196	General Chemistry	NELAP	7/24/2008
Chromium VI	SM 3500-Cr B (20th/21st Ed.)/UV-VIS	General Chemistry	NELAP	9/2/2005
Chrysene	EPA 610	Extractable Organics	NELAP	7/24/2008
Chrysene	EPA 625	Extractable Organics	NELAP	1/24/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Chrysene	EPA 8310	Extractable Organics	NELAP	7/24/2008
cis-1,2-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	7/24/2008
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/24/2008
cis-1,3-Dichloropropene	EPA 601	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 8021	Volatile Organics	NELAP	7/24/2008
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/24/2008
Cobalt	EPA 200.7	Metals	NELAP	1/24/2001
Cobalt	EPA 200.8	Metals	NELAP	1/24/2001
Cobalt	EPA 6010	Metals	NELAP	9/2/2005
Cobalt	EPA 6020	Metals	NELAP	9/2/2005
Color	SM 2120 B	General Chemistry	NELAP	7/24/2008
Conductivity	SM 2510 B	General Chemistry	NELAP	9/2/2005
Copper	EPA 200.7	Metals	NELAP	1/24/2001
Copper	EPA 200.8	Metals	NELAP	1/24/2001
Copper	EPA 6010	Metals	NELAP	9/2/2005
Copper	EPA 6020	Metals	NELAP	9/2/2005
Crotonaldehyde	EPA 8315	Extractable Organics	NELAP	7/24/2008
Cyclohexanone	EPA 8315	Extractable Organics	NELAP	7/24/2008
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Decanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Dibenz(a,h)anthracene	EPA 610	Extractable Organics	NELAP	7/24/2008
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	1/24/2001
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Dibenz(a,h)anthracene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/24/2008
Dibromochloromethane	EPA 601	Volatile Organics	NELAP	1/24/2001
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	1/24/2001

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*Laboratory Scope of Accreditation*

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

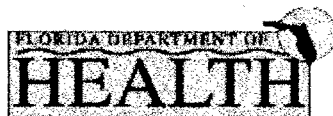
Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibromochloromethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Dibromomethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Dichlorodifluoromethane	EPA 601	Volatile Organics	NELAP	1/24/2001
Dichlorodifluoromethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/24/2008
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/24/2008
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	1/24/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Di-isopropylether (DIPE)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	1/24/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	1/24/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	1/24/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	7/24/2008
Disulfoton	EPA 8270	Extractable Organics	NELAP	7/24/2008
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005

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EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Ethane	RSK-175	Volatile Organics	NELAP	9/2/2005
Ethanol	EPA 8015	Volatile Organics	NELAP	7/24/2008
Ethanol	EPA 8260	Volatile Organics	NELAP	9/2/2005
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	9/2/2005
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	9/2/2005
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Ethylbenzene	EPA 602	Volatile Organics	NELAP	1/24/2001
Ethylbenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Ethylene	RSK-175	Volatile Organics	NELAP	9/2/2005
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Fluoranthene	EPA 610	Extractable Organics	NELAP	7/24/2008
Fluoranthene	EPA 625	Extractable Organics	NELAP	1/24/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Fluoranthene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Fluorene	EPA 610	Extractable Organics	NELAP	7/24/2008
Fluorene	EPA 625	Extractable Organics	NELAP	1/24/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Fluorene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Fluoride	EPA 300.0	General Chemistry	NELAP	5/19/2003
Fluoride	EPA 9056	General Chemistry	NELAP	9/2/2005
Fluoride	SM 4500 F-C	General Chemistry	NELAP	5/19/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	7/24/2008
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	7/24/2008
Hardness	SM 2340 B	General Chemistry	NELAP	7/24/2008
Hardness	SM 2340 C	General Chemistry	NELAP	5/19/2003
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005

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E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	1/24/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	1/24/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Hexachloroethane	EPA 625	Extractable Organics	NELAP	1/24/2001
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	9/2/2005
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Hexanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
Indeno(1,2,3-cd)pyrene	EPA 610	Extractable Organics	NELAP	7/24/2008
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	1/24/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Iron	EPA 200.7	Metals	NELAP	1/24/2001
Iron	EPA 6010	Metals	NELAP	9/2/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8015	Volatile Organics	NELAP	7/24/2008
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Isophorone	EPA 625	Extractable Organics	NELAP	1/24/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	9/2/2005
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	7/24/2008
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Isosafrole	EPA 8270	Extractable Organics	NELAP	9/2/2005
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	1/24/2001
Lead	EPA 200.7	Metals	NELAP	1/24/2001
Lead	EPA 200.8	Metals	NELAP	1/24/2001
Lead	EPA 6010	Metals	NELAP	9/2/2005
Lead	EPA 6020	Metals	NELAP	9/2/2005
Lithium	EPA 200.7	Metals	NELAP	9/2/2005

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lithium	EPA 6010	Metals	NELAP	9/2/2005
m+p-Xylenes	EPA 8260	Volatile Organics	NELAP	7/24/2008
Magnesium	EPA 200.7	Metals	NELAP	1/24/2001
Magnesium	EPA 6010	Metals	NELAP	9/2/2005
Manganese	EPA 200.7	Metals	NELAP	1/24/2001
Manganese	EPA 200.8	Metals	NELAP	1/24/2001
Manganese	EPA 6010	Metals	NELAP	9/2/2005
Manganese	EPA 6020	Metals	NELAP	9/2/2005
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Mercury	EPA 1631	Metals	NELAP	9/9/2005
Mercury	EPA 245.1	Metals	NELAP	5/19/2003
Mercury	EPA 7470	Metals	NELAP	7/24/2008
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	9/2/2005
Methane	RSK-175	Volatile Organics	NELAP	9/2/2005
Methanol	EPA 8015	Volatile Organics	NELAP	7/24/2008
Methapyrilene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Methoxychlor	EPA 608.2	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Methyl bromide (Bromomethane)	EPA 601	Volatile Organics	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 8021	Volatile Organics	NELAP	9/2/2005
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Methyl chloride (Chloromethane)	EPA 601	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 8021	Volatile Organics	NELAP	9/2/2005
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	9/2/2005
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	7/24/2008
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Methylene chloride	EPA 601	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 8021	Volatile Organics	NELAP	9/2/2005
Methylene chloride	EPA 8260	Volatile Organics	NELAP	9/2/2005

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Molybdenum	EPA 200.7	Metals	NELAP	1/24/2001
Molybdenum	EPA 200.8	Metals	NELAP	1/24/2001
Molybdenum	EPA 6010	Metals	NELAP	9/2/2005
Molybdenum	EPA 6020	Metals	NELAP	9/2/2005
Naphthalene	EPA 610	Extractable Organics	NELAP	7/24/2008
Naphthalene	EPA 625	Extractable Organics	NELAP	1/24/2001
Naphthalene	EPA 8021	Volatile Organics	NELAP	9/2/2005
Naphthalene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Naphthalene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Naphthalene	EPA 8310	Extractable Organics	NELAP	7/24/2008
n-Butyl alcohol	EPA 8015	Volatile Organics	NELAP	7/24/2008
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	9/2/2005
n-Butylbenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Nickel	EPA 200.7	Metals	NELAP	1/24/2001
Nickel	EPA 200.8	Metals	NELAP	1/24/2001
Nickel	EPA 6010	Metals	NELAP	9/2/2005
Nickel	EPA 6020	Metals	NELAP	9/2/2005
Nitrate	EPA 9056	General Chemistry	NELAP	9/2/2005
Nitrate as N	EPA 300.0	General Chemistry	NELAP	5/19/2003
Nitrate as N	SM 4500-NO3 F	General Chemistry	NELAP	9/2/2005
Nitrite	EPA 9056	General Chemistry	NELAP	9/2/2005
Nitrite	SM 4500-NO2 B	General Chemistry	NELAP	5/19/2003
Nitrite	SM 4500-NO3 F	General Chemistry	NELAP	9/2/2005
Nitrite as N	EPA 300.0	General Chemistry	NELAP	5/19/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	1/24/2001
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/24/2008
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	1/24/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	1/24/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	1/24/2001

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EPA Lab Code:

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TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/24/2008
Nonanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
n-Propanol	EPA 8015	Volatile Organics	NELAP	7/24/2008
n-Propanol	EPA 8260	Volatile Organics	NELAP	7/24/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/24/2008
Octanal	EPA 8315	Extractable Organics	NELAP	7/24/2008
Oil & Grease	EPA 1664A	General Chemistry	NELAP	1/24/2001
Oil & Grease	EPA 9070	General Chemistry	NELAP	9/2/2005
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	9/2/2005
o-Toluidine	EPA 8270	Extractable Organics	NELAP	9/2/2005
o-Xylene	EPA 8260	Volatile Organics	NELAP	7/24/2008
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Pentachloronitrobenzene (Quintozone)	EPA 8270	Extractable Organics	NELAP	9/2/2005
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	1/24/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
Pentanal (Valeraldehyde)	EPA 8315	Extractable Organics	NELAP	7/24/2008
pH	EPA 9040	General Chemistry	NELAP	7/24/2008
pH	SM 4500-H B	General Chemistry	NELAP	5/19/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	9/2/2005
Phenanthrene	EPA 610	Extractable Organics	NELAP	7/24/2008
Phenanthrene	EPA 625	Extractable Organics	NELAP	1/24/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Phenanthrene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Phenol	EPA 625	Extractable Organics	NELAP	1/24/2001
Phenol	EPA 8270	Extractable Organics	NELAP	9/2/2005
Phorate	EPA 8270	Extractable Organics	NELAP	9/2/2005
Phosphorus, total	SM 4500-P F	General Chemistry	NELAP	7/24/2008
p-Isopropyltoluene	EPA 8021	Volatile Organics	NELAP	7/24/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	9/2/2005

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Potassium	EPA 200.7	Metals	NELAP	1/24/2001
Potassium	EPA 6010	Metals	NELAP	9/2/2005
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	9/2/2005
Propanal (Propionaldehyde)	EPA 8315	Extractable Organics	NELAP	7/24/2008
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Pyrene	EPA 610	Extractable Organics	NELAP	7/24/2008
Pyrene	EPA 625	Extractable Organics	NELAP	1/24/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	9/2/2005
Pyrene	EPA 8310	Extractable Organics	NELAP	7/24/2008
Pyridine	EPA 8270	Extractable Organics	NELAP	7/24/2008
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/24/2008
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	9/2/2005
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	9/2/2005
Residue-settleable	SM 2540 F	General Chemistry	NELAP	7/24/2008
Residue-total	SM 2540 B	General Chemistry	NELAP	9/2/2005
Residue-volatile	EPA 160.4	General Chemistry	NELAP	1/24/2001
Safrole	EPA 8270	Extractable Organics	NELAP	9/2/2005
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Selenium	EPA 200.7	Metals	NELAP	1/24/2001
Selenium	EPA 200.8	Metals	NELAP	1/24/2001
Selenium	EPA 6010	Metals	NELAP	9/2/2005
Selenium	EPA 6020	Metals	NELAP	9/2/2005
Silicon	EPA 200.7	Metals	NELAP	1/24/2001
Silicon	EPA 6010	Metals	NELAP	9/2/2005
Silver	EPA 200.7	Metals	NELAP	1/24/2001
Silver	EPA 200.8	Metals	NELAP	1/24/2001
Silver	EPA 6010	Metals	NELAP	9/2/2005
Silver	EPA 6020	Metals	NELAP	9/2/2005
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Sodium	EPA 200.7	Metals	NELAP	1/24/2001
Sodium	EPA 6010	Metals	NELAP	9/2/2005
Strontium	EPA 200.7	Metals	NELAP	9/2/2005
Strontium	EPA 6010	Metals	NELAP	7/24/2008
Styrene	EPA 8021	Volatile Organics	NELAP	7/24/2008
Styrene	EPA 8260	Volatile Organics	NELAP	7/24/2008
Sulfate	ASTM D516-02	General Chemistry	NELAP	7/24/2008

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sulfate	ASTM D516-90	General Chemistry	NELAP	7/24/2008
Sulfate	EPA 300.0	General Chemistry	NELAP	5/19/2003
Sulfate	EPA 9038	General Chemistry	NELAP	9/2/2005
Sulfate	EPA 9056	General Chemistry	NELAP	9/2/2005
Sulfide	SM 4500-S D/UV-VIS	General Chemistry	NELAP	5/19/2003
Sulfide	SM 4500-S F	General Chemistry	NELAP	9/2/2005
Sulfite-SO3	SM 4500-SO3 B	General Chemistry	NELAP	5/19/2003
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	5/19/2003
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/24/2008
tert-Butylbenzene	EPA 8021	Volatile Organics	NELAP	9/2/2005
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Tetrachloroethylene (Perchloroethylene)	EPA 601	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8021	Volatile Organics	NELAP	9/2/2005
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/24/2008
Thallium	EPA 200.7	Metals	NELAP	1/24/2001
Thallium	EPA 200.8	Metals	NELAP	1/24/2001
Thallium	EPA 6010	Metals	NELAP	9/2/2005
Thallium	EPA 6020	Metals	NELAP	9/2/2005
Thionazin (Zinophos)	EPA 8270	Extractable Organics	NELAP	7/24/2008
Tin	EPA 200.7	Metals	NELAP	9/2/2005
Tin	EPA 6010	Metals	NELAP	9/2/2005
Tin	EPA 6020	Metals	NELAP	9/2/2005
Tin (limited CWA use only per EPA ATP)	EPA 200.8	Metals	NELAP	7/24/2008
Titanium	EPA 200.7	Metals	NELAP	9/2/2005
Titanium	EPA 6010	Metals	NELAP	9/2/2005
Toluene	EPA 602	Volatile Organics	NELAP	1/24/2001
Toluene	EPA 624	Volatile Organics	NELAP	1/24/2001
Toluene	EPA 8021	Volatile Organics	NELAP	9/2/2005
Toluene	EPA 8260	Volatile Organics	NELAP	9/2/2005
Total cyanide	EPA 9014	General Chemistry	NELAP	7/24/2008
Total cyanide	SM 4500CN-E	General Chemistry	NELAP	9/2/2005
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	5/19/2003
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	9/2/2005

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EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total nitrate-nitrite	SM 4500-NO3 F	General Chemistry	NELAP	9/2/2005
Total organic carbon	SM 5310 C	General Chemistry	NELAP	7/24/2008
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	7/24/2008
Total phenolics	EPA 420.1	General Chemistry	NELAP	7/24/2008
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/2/2005
trans-1,2-Dichloroethylene	EPA 601	Volatile Organics	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	9/2/2005
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	9/2/2005
trans-1,3-Dichloropropylene	EPA 601	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropylene	EPA 8021	Volatile Organics	NELAP	9/2/2005
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	9/2/2005
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/24/2008
Trichloroethene (Trichloroethylene)	EPA 601	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 8021	Volatile Organics	NELAP	9/2/2005
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Trichlorofluoromethane	EPA 601	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 8021	Volatile Organics	NELAP	9/2/2005
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	9/2/2005
Turbidity	SM 2130 B	General Chemistry	NELAP	9/2/2005
Uranium	EPA 200.8	Metals	NELAP	7/24/2008
Uranium	EPA 6020	Metals	NELAP	7/24/2008
Vanadium	EPA 200.7	Metals	NELAP	1/24/2001
Vanadium	EPA 200.8	Metals	NELAP	1/24/2001
Vanadium	EPA 6010	Metals	NELAP	9/2/2005
Vanadium	EPA 6020	Metals	NELAP	9/2/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/24/2008
Vinyl chloride	EPA 601	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 8021	Volatile Organics	NELAP	9/2/2005
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	9/2/2005
Xylene (total)	EPA 602	Volatile Organics	NELAP	5/19/2003

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

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TriMatrix Laboratories, Inc.

5560 Corporate Exchange Court SE

Grand Rapids, MI 49512

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Xylene (total)	EPA 624	Volatile Organics	NELAP	5/19/2003
Xylene (total)	EPA 8021	Volatile Organics	NELAP	9/2/2005
Xylene (total)	EPA 8260	Volatile Organics	NELAP	9/2/2005
Zinc	EPA 200.7	Metals	NELAP	1/24/2001
Zinc	EPA 200.8	Metals	NELAP	1/24/2001
Zinc	EPA 6010	Metals	NELAP	9/2/2005
Zinc	EPA 6020	Metals	NELAP	9/2/2005

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





# *Laboratory Scope of Accreditation*

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.

5560 Corporate Exchange Court SE

Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8021	Volatile Organics	NELAP	5/19/2003
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/19/2003
1,1,1-Trichloroethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1,2,2-Tetrachloroethane	EPA 8021	Volatile Organics	NELAP	5/19/2003
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/19/2003
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1,1,2-Trichloroethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,2,4-Trichlorobenzene	EPA 8021	Volatile Organics	NELAP	5/19/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dichloropropane	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/24/2008
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	1/24/2001

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	1/24/2001
1,3-Tolualdehyde	EPA 8315	Extractable Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	1/24/2001
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/24/2008
1-Methylnaphthalene (added to method at FDEP request)	EPA 8270	Extractable Organics	NELAP	7/24/2008
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	1/24/2001
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	1/24/2001
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	5/19/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	5/19/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	1/24/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	1/24/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Chloroethyl vinyl ether	EPA 8021	Volatile Organics	NELAP	1/24/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	1/24/2001

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	5/19/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Hexanone	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	1/24/2001
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	5/19/2003
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	1/24/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	1/24/2001
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	1/24/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	1/24/2001
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	1/24/2001
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	1/24/2001
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	1/24/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	1/24/2001
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	5/19/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	5/19/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	1/24/2001
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	1/24/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	1/24/2001
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	1/24/2001

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011





### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Acenaphthene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	1/24/2001
Acetaldehyde	EPA 8315	Extractable Organics	NELAP	1/24/2001
Acetone	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acetophenone	EPA 8270	Extractable Organics	NELAP	1/24/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	1/24/2001
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/24/2008
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Aluminum	EPA 6010	Metals	NELAP	1/24/2001
Aniline	EPA 8270	Extractable Organics	NELAP	1/24/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Anthracene	EPA 8310	Extractable Organics	NELAP	1/24/2001
Antimony	EPA 6010	Metals	NELAP	1/24/2001
Antimony	EPA 6020	Metals	NELAP	1/24/2001
Aramite	EPA 8270	Extractable Organics	NELAP	1/24/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Arsenic	EPA 6010	Metals	NELAP	1/24/2001
Arsenic	EPA 6020	Metals	NELAP	1/24/2001
Barium	EPA 6010	Metals	NELAP	1/24/2001
Barium	EPA 6020	Metals	NELAP	1/24/2001
Benzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Benzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Benzidine	EPA 8270	Extractable Organics	NELAP	1/24/2001

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*Laboratory Scope of Accreditation*

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	1/24/2001
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	1/24/2001
Beryllium	EPA 6010	Metals	NELAP	1/24/2001
Beryllium	EPA 6020	Metals	NELAP	1/24/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	5/19/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	5/19/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/24/2008
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	1/24/2001
Boron	EPA 6010	Metals	NELAP	1/24/2001
Bromobenzene	EPA 8021	Volatile Organics	NELAP	7/24/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 8021	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	1/24/2001
Butanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	5/19/2003
Cadmium	EPA 6010	Metals	NELAP	1/24/2001
Cadmium	EPA 6020	Metals	NELAP	1/24/2001
Calcium	EPA 6010	Metals	NELAP	1/24/2001
Carbazole	EPA 8270	Extractable Organics	NELAP	5/19/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 8021	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	1/24/2001

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Chloride	EPA 9056	General Chemistry	NELAP	5/19/2003
Chlorobenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chlorobenzilate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Chloroethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 8021	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chloroprene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chromium	EPA 6010	Metals	NELAP	1/24/2001
Chromium	EPA 6020	Metals	NELAP	1/24/2001
Chromium VI	EPA 7196	General Chemistry	NELAP	5/19/2003
Chrysene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Chrysene	EPA 8310	Extractable Organics	NELAP	1/24/2001
cis-1,2-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	1/24/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 8021	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Cobalt	EPA 6010	Metals	NELAP	1/24/2001
Cobalt	EPA 6020	Metals	NELAP	1/24/2001
Copper	EPA 6010	Metals	NELAP	1/24/2001
Copper	EPA 6020	Metals	NELAP	1/24/2001
Crotonaldehyde	EPA 8315	Extractable Organics	NELAP	1/24/2001
Cyclohexanone	EPA 8315	Extractable Organics	NELAP	1/24/2001
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Decanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Diallate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Dibenz(a,h)anthracene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	1/24/2001
Dibromochloromethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibromomethane	EPA 8021	Volatile Organics	NELAP	7/24/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Dichlorodifluoromethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	5/19/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	1/24/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	1/24/2001
Di-isopropylether (DIPE)	EPA 8260	Volatile Organics	NELAP	7/24/2008
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	1/24/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	5/19/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	5/19/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Diphenylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Ethanol	EPA 8015	Volatile Organics	NELAP	1/24/2001
Ethanol	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	1/24/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Fluoranthene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Fluorene	EPA 8310	Extractable Organics	NELAP	5/19/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Fluoride	EPA 9056	General Chemistry	NELAP	5/19/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	1/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	5/19/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Heptanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/19/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	7/24/2008
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	5/19/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Hexanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
Ignitability	EPA 1020	General Chemistry	NELAP	1/24/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Iron	EPA 6010	Metals	NELAP	1/24/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8015	Volatile Organics	NELAP	1/24/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Isodrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	5/19/2003
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	1/24/2001
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Isosafrole	EPA 8270	Extractable Organics	NELAP	1/24/2001
Kepone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Lead	EPA 6010	Metals	NELAP	1/24/2001
Lead	EPA 6020	Metals	NELAP	1/24/2001
Lithium	EPA 6010	Metals	NELAP	1/24/2001
m+p-Xylenes	EPA 8260	Volatile Organics	NELAP	9/9/2005

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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Attachment to Certificate #: E87622-13, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87622

EPA Lab Code:

MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Magnesium	EPA 6010	Metals	NELAP	1/24/2001
Manganese	EPA 6010	Metals	NELAP	1/24/2001
Manganese	EPA 6020	Metals	NELAP	1/24/2001
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Mercury	EPA 7471	Metals	NELAP	1/24/2001
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methanol	EPA 8015	Volatile Organics	NELAP	1/24/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 8021	Volatile Organics	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 8021	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	1/24/2001
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/19/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 8021	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	1/24/2001
Molybdenum	EPA 6010	Metals	NELAP	1/24/2001
Molybdenum	EPA 6020	Metals	NELAP	7/24/2008
Naphthalene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Naphthalene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Naphthalene	EPA 8310	Extractable Organics	NELAP	1/24/2001
n-Butyl alcohol	EPA 8015	Volatile Organics	NELAP	1/24/2001
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	1/24/2001
n-Butylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Nickel	EPA 6010	Metals	NELAP	1/24/2001
Nickel	EPA 6020	Metals	NELAP	1/24/2001
Nitrate	EPA 9056	General Chemistry	NELAP	5/19/2003
Nitrite	EPA 9056	General Chemistry	NELAP	5/19/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	5/19/2003

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Expiration Date: 6/30/2011



*Laboratory Scope of Accreditation*

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

**E87622**

**TriMatrix Laboratories, Inc.**  
**5560 Corporate Exchange Court SE**  
**Grand Rapids, MI 49512**

**Matrix: Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/24/2008
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	5/19/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
Nonanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
n-Propanol	EPA 8260	Volatile Organics	NELAP	7/24/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	1/24/2001
Octanal	EPA 8315	Extractable Organics	NELAP	1/24/2001
Oil & Grease	EPA 9071	General Chemistry	NELAP	1/24/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	1/24/2001
o-Xylene	EPA 8260	Volatile Organics	NELAP	9/9/2005
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	1/24/2001
Paraldehyde	EPA 8015	Volatile Organics	NELAP	1/24/2001
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
p-Dioxane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	1/24/2001
Pentachloronitrobenzene (Quintozone)	EPA 8270	Extractable Organics	NELAP	1/24/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	1/24/2001
Pentanal (Valeraldehyde)	EPA 8315	Extractable Organics	NELAP	1/24/2001
pH	EPA 9045	General Chemistry	NELAP	1/24/2001
Phenacetin	EPA 8270	Extractable Organics	NELAP	1/24/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Phenanthrene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Phenol	EPA 8270	Extractable Organics	NELAP	5/19/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Potassium	EPA 6010	Metals	NELAP	1/24/2001

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Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

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State Laboratory ID: E87622

EPA Lab Code: MI00005

(616) 975-4500

E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	1/24/2001
Propanal (Propionaldehyde)	EPA 8315	Extractable Organics	NELAP	1/24/2001
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	5/19/2003
Pyrene	EPA 8310	Extractable Organics	NELAP	5/19/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	1/24/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	1/24/2001
Safrole	EPA 8270	Extractable Organics	NELAP	1/24/2001
sec-Butylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Selenium	EPA 6010	Metals	NELAP	1/24/2001
Selenium	EPA 6020	Metals	NELAP	7/24/2008
Silicon	EPA 6010	Metals	NELAP	1/24/2001
Silver	EPA 6010	Metals	NELAP	1/24/2001
Silver	EPA 6020	Metals	NELAP	1/24/2001
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/24/2008
Sodium	EPA 6010	Metals	NELAP	1/24/2001
Strontium	EPA 6010	Metals	NELAP	1/24/2001
Styrene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Styrene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Sulfate	EPA 9038	General Chemistry	NELAP	7/24/2008
Sulfate	EPA 9056	General Chemistry	NELAP	5/19/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	1/24/2001
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	1/24/2001
tert-Butyl alcohol	EPA 8015	Volatile Organics	NELAP	1/24/2001
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	1/24/2001
tert-Butylbenzene	EPA 8021	Volatile Organics	NELAP	1/24/2001
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8021	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	1/24/2001
Thallium	EPA 6010	Metals	NELAP	1/24/2001
Thallium	EPA 6020	Metals	NELAP	1/24/2001
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	1/24/2001
Tin	EPA 6010	Metals	NELAP	1/24/2001

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Governor



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### Laboratory Scope of Accreditation

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EPA Lab Code: MI00005

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E87622

TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Tin	EPA 6020	Metals	NELAP	7/24/2008
Titanium	EPA 6010	Metals	NELAP	5/19/2003
Toluene	EPA 8021	Volatile Organics	NELAP	1/24/2001
Toluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Total cyanide	EPA 9014	General Chemistry	NELAP	9/2/2005
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	5/19/2003
Total organic carbon	WALKLEY-BLACK	General Chemistry	NELAP	9/2/2005
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	1/24/2001
Total phenolics	EPA 9065	General Chemistry	NELAP	7/24/2008
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 8021	Volatile Organics	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropylene	EPA 8021	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 8021	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 8021	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Trifluralin (Treflan)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/19/2003
Vanadium	EPA 6010	Metals	NELAP	1/24/2001
Vanadium	EPA 6020	Metals	NELAP	7/24/2008
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 8021	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	1/24/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	1/24/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Zinc	EPA 6010	Metals	NELAP	1/24/2001
Zinc	EPA 6020	Metals	NELAP	1/24/2001

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



### **Attachment 3**

Laboratory Quality Assurance Manual





# **Quality Assurance Manual**

## **Analytical Services**

**Release Date December, 2008**

*Prepared by:*  
TriMatrix Laboratories, Inc.  
5560 Corporate Exchange Court  
Grand Rapids, MI 49512  
616-975-4500



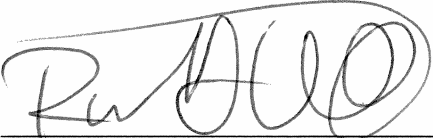
## QUALITY ASSURANCE MANUAL

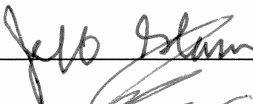
### Policies and Procedures Required of the Personnel Employed by TriMatrix Laboratories, Inc., Including the Organic, Inorganic, and Metals Laboratory Areas

Revision Number: 7.0

Effective Date: December 2008


**Initial Approvals:**

Quality Assurance Manager:  Date: 12/2/08

Technical Director:  Date: 12/5/08

Laboratory President:  Date: 12/4/08

**Subsequent Approvals:**

Quality Assurance Manager:  Date: 2/16/10

Quality Assurance Manager: \_\_\_\_\_ Date: \_\_\_\_\_

Quality Assurance Manager: \_\_\_\_\_ Date: \_\_\_\_\_

Quality Assurance Manager: \_\_\_\_\_ Date: \_\_\_\_\_

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## **8.0 GLOSSARY OF TERMS**



### **3.0 QUALITY SYSTEM**

#### **3.1 INTRODUCTION: THE TriMatrix QUALITY SYSTEM**

##### **3.1.1 Manual Purpose**

The purpose of this manual is to outline the organization, specify the procedures, and define the technical requirements utilized by TriMatrix Laboratories, Inc. The goal is to ensure that all data generated is of the required quality, is reproducible, and is generated in a timely manner. This manual details a Quality Assurance/Quality Control (QA/QC) program encompassing the entire analytical efforts at TriMatrix, from project initiation to report generation. Some areas are covered with only a cursory discussion, while others are covered in detail, or are included in more than one section, depending on their importance. This manual describes the realistic functions of the quality programs in place, with an understanding that not every situation is covered nor every contingency explored.

##### **3.1.2 The Need for Analytical Quality Assurance/Quality Control**

In the increasingly competitive business of environmental laboratory services, the primary tenet of continued success is to efficiently provide results of the necessary quality. TriMatrix agrees with this tenet, considers analytical quality assurance and quality control to be of prime importance, and has incorporated it as the central pillar of our efforts to remain on the leading edge of the environmental laboratory field. The requirements we place on ourselves are in concert with the needs and agendas of other organizations, such as the Environmental Protection Agency (EPA), governmental and industrial clients, and various state and local regulatory agencies.

Quality assurance and quality control (QA/QC) functions absorb nearly fifty percent of the available effort involved in routine analysis, and continues to evolve and grow in importance. This level of quality is absolutely essential for two reasons: 1) accurate analytical data is obtained only with the concurrent



use of extensive QA/QC to regulate and monitor the many process variables that can potentially introduce errors into chemical analyses, and 2) clients make crucial business decisions based on the data supplied by the laboratory. Lab data not properly supported by adequate quality assurance/quality control practices and procedures can be questionable at best, and can lead to faulty or erroneous decisions in the field. In the overall analytical effort the additional time spent for QA/QC is time necessarily spent.

### **3.1.3 Definition of Terms**

#### **3.1.3.1 Quality Assurance**

Quality Assurance (QA) is defined as those operations and procedures undertaken to provide measurement data of documentable quality that have a stated probability of being accurate. The measurement system part of the quality assurance program must be in statistical control to justify this probability statement.

The operations and procedures established as part of the overall quality assurance program encompass all aspects of the laboratory operations, including but not limited to: organizational structure, human resources, physical resources, methodology, analyst training and certification, data reduction, data validation, and instrument maintenance and troubleshooting. All aspects of QA are organized, implemented, and monitored through written standard operating procedures.

#### **3.1.3.2 Quality Control**

Quality control is defined as the basic checks necessary to produce a good measurement program. These checks include but are not limited to: proper calibration and calibration verification, statistical monitoring of accuracy and precision, of quality control samples



(e.g. laboratory control samples, blanks, duplicates, spikes, etc.), interference monitoring, and reagent control.

Adequate records are maintained to support data quality, to locate assignable causes in measurement problems, to improve the accuracy and precision of the measurement system, and to provide a historical record of traceability.

### **3.1.3.3 Quality Assessment**

Quality assessment is defined as those specific steps utilized to evaluate the quality of the measurement process. These steps include use of control charts to plot multiple data points over time, monitoring parameters by statistical control, internal performance audits, external performance audits, certification programs conducted by individual states, and performance evaluation sample programs.

## **3.2 QUALITY POLICY STATEMENTS FROM MANAGEMENT**

As communicated from top management through the entire organization, TriMatrix Laboratories, Inc. is driven by the following quality objectives and commitments.

### **3.2.1 Corporate Quality Objectives**

- To create and maintain a uniform and controlled pattern for performing routine tasks within the organization, based on standard operating procedures.
- To generate legally defensible, scientifically sound laboratory data of documented quality.
- To build quality into the workplace ensuring services contributing to successful relationships with our customers, employees, and vendors.



- To develop, deliver, and maintain, excellence in all operational areas.
- To provide a service that consistently meets or exceeds client expectations.

### **3.2.2 Corporate Quality Commitments**

- To support quality by underwriting the substantial cost of the quality commitment even though such expenses do not result in increased productivity or a tangible product.
- To maintain a work environment in which all employees are free from commercial pressures in the performance of their duties.
- To maintain a work environment in which all employees are free from internal organization or external client related pressures that may influence the quality of their work.
- To educate all employees in fraud prevention and their ethical responsibilities associated with analytical and data reporting activities.
- To ensure that client confidentiality and information are strictly protected.
- To implement on-going improvement in every area of laboratory activity.
- To create and maintain a Quality Environment with an all-encompassing determination to meet the needs and quality objectives of our clients.
- To commit and adhere to the requirements specified in ISO/IEC 17025.
- To commit and adhere to the requirements specified by the NELAC Standards.



Included with these improvements and commitments is an annual review process where the management of TriMatrix Laboratories performs a comprehensive review of the quality system. This review monitors the effectiveness of the quality system and provides feedback for on-going improvement. Policy changes made as a result of the annual review will be reflected in the QA Manual.

### **3.3 ORGANIZATION AND RESPONSIBILITIES**

An efficient organizational operation requires a quality control program facilitating a high level of multi-directional communication and information flow. Each person in the TriMatrix organization inputs and receives information from the quality system. This information flow optimizes management directives with minimum disruption, and provides the means for creating improvements.

#### **3.3.1 Corporate Structure**

Flow of both administrative and quality control information is presented in Figure 3-1. This diagram graphically displays the corporate philosophy concerning the interaction of QA/QC and the generation of analytical data. The general flow of data in this format gives QA/QC independence in fulfilling its function while still acting as a liaison with the administrative staff. To further explain this interaction, a detailed description of roles and responsibilities is presented for each key laboratory position.

#### **3.3.2 Laboratory President**

Responsibilities of the Laboratory President are directed at the overall operation and management of the laboratory. Primary responsibilities include, but are not limited to: 1) develop and meet budgets established for the laboratory, 2) manage analytical services productivity and quality, 3) oversee and develop new business activities including client relations development, 4) plan analytical services organization, leadership and management programs, 5)



develop and manage human resources including career path planning, and 6) performing duties as Deputy Technical Director when necessary.

### **3.3.3 Quality Assurance Manager**

The Quality Assurance Manager is primarily responsible for the implementation, maintenance, reporting, and development of all QA/QC activities performed within the laboratory. Duties include, but are not limited to: 1) QA/QC systems development and monitoring, 2) coordination of all documentation procedures including the development and control of standard operation procedures, 3) monitoring method and quality control requirements as published by regulatory agencies ISO/IEC 17025, and the NELAC Standards, 4) performing internal lab audits, 5) maintaining in-house QA/QC monitoring procedures and policies, and 6) providing quality assurance guidance and training to all staff members. The Quality Assurance Manager has the authority to stop work as a result of poor data quality.

### **3.3.4 Technical Director**

The Technical Director is responsible for the overall technical capabilities and direction of the laboratory. Specific responsibilities include: 1) organization and management of new analytical technologies developed by the laboratory, 2) adherence to ISO/IEC 17025 requirements and NELAC Standards, 3) equipment procurement management.

### **3.3.5 Health and Safety Officer**

The Health and Safety Officer is responsible for implementation, monitoring, and maintenance of all laboratory safety and chemical hygiene programs. Specific responsibilities include the development and maintenance of health and safety programs and manuals.

### **3.3.6 Vice President of Laboratory Operations**



The Vice President of Laboratory Operations is responsible for the overall supervision of the individual laboratory areas. General responsibilities include management of staff activities such as scheduling, budgeting, training, and general supervision. The Vice President of Laboratory Operations also is responsible for 1) the development and management of all chemists, analysts, technicians, 2) implementation of quality systems and controls within the laboratory, 3) scheduling analysis activities, 4) meeting productivity goals and project deadlines, 5) technical development of the laboratory staff, 6) approval of laboratory's SOPs, 7) coordination of methods development with the staff and Technical Director, 8) approval of laboratory data, or the delegation thereof, 9) Approval of procurement activities, 10) Overall laboratory performance, and 11) adherence to ISO/IEC 17025 requirements.

### **3.3.7 Client Services Manager**

The laboratory Client Services Manager supervises both the Client Services and the Data Management Group. Responsibilities of the Client Services Manager include management of scheduling and method development needs, budgeting, training, and general supervision, with specific emphasis on the following activities: 1) development and management of all project chemists, project chemist technicians, log-in staff, bottle preparation staff, laboratory couriers, the Field Services Group, and Data Management Group, 2) project management, 3) coordination of proposal preparation and marketing activities for existing and new clients, 4) monitoring of final report turnaround times and, 5) monitoring client satisfaction with laboratory services.

### **3.3.8 Deputy Quality Assurance Manager/Deputy Technical Director**

The Deputy Quality Assurance Manager/Technical Director has the responsibility of fulfilling an interim role as outlined in sections 3.3.3, 3.3.4, 3.5.1.2, and 3.5.1.3.

### **3.3.9 Sales and Marketing Staff**



The Sales and Marketing Staff are responsible for all marketing, business development, and client maintenance activities. These activities include but are not necessarily limited to: 1) market research/gathering market intelligence, 2) consulting with company management to develop a corporate business strategy and plan, 3) development and implementation of a corporate image campaign, 4) development and distribution of marketing materials (corporate literature, etc.), 5) client prospecting, 6) presenting/introducing company services to prospective clients, 7) account development, management and maintenance (in conjunction with Project Chemists), 8) development of corporate pricing guidelines, 9) development of proposals, quotations, bids and qualifications summaries, and 10) contract review, negotiation and execution.

#### **3.3.10 Organizational Chart**

Presented in Figure 3-2 is an organizational chart illustrating the personnel structure within the laboratory.

### **3.4 RELATIONSHIPS**

Relationships within the analytical laboratory are organized through management into three main categories: Technical Operations, Support Services, and the Laboratory Quality System. The relationships between management and these operations define and maintain the delicate balance in a cost-effective, highly-technical, quality laboratory operation. An overview of each relation is presented below:

#### **3.4.1 Management-Technical Operations**

The relationship between management and technical operations is illustrated in Figure 3-3. In this relationship, the main role of management is to provide guidance and financial support to the programs and directives of the Technical Director. Through this structure, technical operational enhancements and developments occur and are applied through the laboratory staff.

#### **3.4.2 Management-Support Services**



The relationship between management and support services is illustrated in Figure 3-4. In this relationship, management's role is substantial in the day-to-day operation of each service.

The primary laboratory support groups are Client Services, Sales and Marketing, and LIMS system support. These groups report directly to the Laboratory President for all aspects of their daily activities.

Secondary relationships are maintained with the Laboratory Administrative Assistant, Laboratory Receptionist, Accounting, and the Human Resources Department. Some groups within this secondary category maintain relationships not only with the Laboratory President, but also with other management groups within the TriMatrix organization.

A tertiary relationship has been developed between the Laboratory President and Vice President of Laboratory Operations. This relationship supports productivity monitoring, cost containment, equipment procurement, operations management, personnel/human resources activities, technical support, data validation, and method development.

### **3.4.3 Management-Quality System**

The relationship between management and the laboratory quality system is illustrated in Figure 3-5. In this relationship, management plays a secondary role in the overall scheme. This secondary role provides the quality assurance manager with guidance, company perspective, and structured support in the development, implementation, and maintenance of quality system programs and activities.

This relationship is vital to the success of TriMatrix Laboratories. Without a cost-effective quality system, the overall caliber of laboratory data and the success of all laboratory operations would be jeopardized.



A relationship also exists between management, the quality system, the laboratory support, and the HR staff. This relationship includes but is not limited to: laboratory management directives, and human resources/personnel activities. These activities are implemented and maintained without disruption to the quality system, and are depicted via the dashed lines on Figure 3-5.

### **3.5 JOB DESCRIPTIONS**

The strength of a laboratory lies in the experience and dedication of its employees. TriMatrix hires quality personnel based both on work attitude and past job experience. Job descriptions have been written to define the employee qualifications required for each position.

#### **3.5.1 Management Staff Members**

Managerial positions are responsible for the development of their respective employees. These positions have specific minimum requirements for years of experience.

##### **3.5.1.1 Laboratory President**

###### **Job Description**

The Laboratory President (LP) directs the laboratory. The LP works through the Vice President of Laboratory Operations to improve data quality, overall productivity, staff development, safety/training programs, and overall profitability. This position has profit/loss accountability. Budgets are developed annually with senior management. The LP is also directly involved in business development/sales activities, and the sales staff reports directly to him.

###### **Background/Educational Requirements**

The LP possesses minimally a bachelor's degree in science, preferably chemistry. The LP has a minimum of 10 years direct



work experience in the environmental testing industry. This work experience includes having conducted environmental analyses and several years of demonstrated supervisory experience.

**Duties and Responsibilities**

1. Development and fulfillment of budgets.
2. Management of total laboratory productivity and quality.
3. Management of proposal preparation.
4. Development of new business and maintenance of client relationships.
5. Development of laboratory organization, leadership, and management planning.
6. Working with the Human Resources department to develop staff members and their career paths.

**3.5.1.2      Quality Assurance Manager**

**Job Description**

The Quality Assurance (QA) Manager is responsible for the development, implementation, improvement, and maintenance of all quality systems at TriMatrix. The QA Manager monitors all the analytical methods and procedures performed by the laboratory, and assures compliance with regulatory agency requirements.

**Background/Educational Requirements**

The QA Manager possesses a B.S. in science, preferably chemistry, and suitable work experience. Work experience must include several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of quality assurance and statistical quality control procedures, specifically as they apply to analytical protocols, is required.

**Duties and Responsibilities**



1. Development and implementation of systems to measure and monitor laboratory data quality.
2. Maintenance of the documentation system for generation, control, and archiving laboratory forms, SOPs, and protocols.
3. Approving SOPs and monitoring their compliance with regulatory agency requirements.
4. Maintaining and updating the laboratory Quality Assurance Manual.
5. On-going investigation for optimizing procedures to minimize out-of-control data.
6. Maintenance of federal, state, and industrial certifications and accreditations as required.
7. Monitoring internal quality programs within the laboratory and reporting their status to management.
8. Training and training documentation of all staff members in all aspects of the laboratory quality system.
9. Perform other duties as deemed necessary by management.

### **3.5.1.3 Technical Director**

#### **Job Description**

The Technical Director (TD) is responsible for the development and improvement of technical operations within the laboratory division. The TD oversees the investigation of all new instruments and equipment, method development, and general technical advancement of the laboratory. The TD is also responsible for informing the Deputy TD of current and pending projects and activities.

#### **Background/Educational Requirements**

The TD possesses a B.S. in science, preferably chemistry, and suitable work experience. Such work experience includes several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of



instruments and methodologies, specifically as they apply to analytical protocols, is required.

#### **Duties and Responsibilities**

1. On-going technical development of the TriMatrix Laboratory pertaining to current and future analytical practices.
2. Overseeing the technical development of TriMatrix staff in the areas of method comprehension and implementation.
3. Development of new analytical procedures within the laboratory.
4. Providing technical advice regarding all equipment and apparatus procurement, and acquisitions.
5. Performing technical review of all Quality Assurance Project Plans (QAPPs).
6. Perform other duties as deemed necessary by management.

#### **3.5.1.4 Client Services Manager**

##### **Job Description**

The Client Services (CS) Manager is responsible for the supervision of the project chemists, project chemist technicians, sample log-in staff, bottle preparation staff, laboratory couriers, field services group, and laboratory administrative staff. These responsibilities include meeting project due dates, preparing and reviewing quotations, project initiation and management, client satisfaction management, and supervision and training of staff. The CS Manager strives for improvement in the on-time delivery of laboratory projects.

##### **Background/Educational Requirements**

The CS Manager possesses a B.S. in science, preferably chemistry, and has 5-10 years of work experience. The work experience includes 3-5 years of laboratory experience, involvement in client



management activities, and a demonstrated ability to supervise and train laboratory staff.

**Duties and Responsibilities**

1. Responsible for the productivity and quality of the client services group.
2. Management of large Level 3 or higher projects.
3. Quality control program implementation and maintenance.
4. Supervision and technical development of employees.
5. Development and maintenance of standard operating procedures.
6. Assisting and coordinating marketing activities through proposal preparation and client visitation.
7. Perform other duties as deemed necessary by management.

**3.5.1.5 Vice President of Laboratory Operations**

**Job Description**

The Vice President of Laboratory Operations (VPLO) is responsible for the individual laboratory areas and the supervision of laboratory staff. These responsibilities include meeting project schedules, and the supervision and training of staff members. The VPLO continually works to improve the quality of data generated.

**Background/Educational Requirements**

The VPLO possesses a B.S. degree in science, preferably chemistry, and 5-10 years work experience. The work experience includes a minimum of 5 years in the laboratory utilizing a variety of techniques. The VPLO must also demonstrate an ability to supervise and train staff members.

**Duties and Responsibilities**

1. Responsible for the productivity and quality of the laboratory areas.



2. Operation and maintenance of instrumentation and apparatus.
3. Quality control program implementation and maintenance.
4. Reviewing and final approval of all organic data.
5. Scheduling in-house to allow on-time report generation.
6. Supervision of supply acquisition activities.
7. Supervision and technical development of employees.
8. Approval of standard operating procedures.
9. Methods development.
10. Perform other duties as deemed necessary by management.

#### **3.5.1.6 Laboratory Computer Systems Administrator**

##### **Job Description**

Provide technical review, guidance, and training in current and future laboratory computer applications.

##### **Background/Educational Requirements**

Requires a degree in computer sciences with an emphasis in a chemistry or general science curriculum.

##### **Duties and Responsibilities**

1. Developing a complete understanding of the Laboratory Information Management System (LIMS).
2. Reviewing laboratory computer applications and processes, including instrument computer interfaces, data transmission/archiving processes and document control.
3. Providing database maintenance support activities for the LIMS system.
4. Providing technical direction and orchestrating implementation of electronic storage systems for the laboratory.
5. Providing technical training of the laboratory staff in software applications and basic computer operational activities.
6. Perform other duties as deemed necessary by management.



### **3.5.2 Technical Staff Members**

Technical staff members are classified into chemist or technician levels dependant on job type, education, and years of experience. Level Classifications are Chemist I-V and Senior Chemist, Project Chemist I-V and Senior Project Chemist, Technician I-V and Senior Technician. In addition, qualified candidates are also eligible for group leader status. Classification descriptions are provided in Appendix A. To aid the employee in identifying the different classification requirements, the differences are printed in bold italicized text. The various classifications are also used by the employee and by management for career path development at TriMatrix.

## **3.6 MANAGEMENT RESUMES**

Laboratory President

Quality Assurance Manager

Vice President of Laboratory Operations

Human Resources Manager



**DOUGLAS E. KRISCUNAS**

**Laboratory President**

**EDUCATION**

B.S., Environmental Sciences, Grand Valley State University, 1976

**PROFESSIONAL SUMMARY**

Mr. Kriscunas is responsible for the accuracy and integrity of all analytical data finalized at this location. He is continuously available for client support to resolve analytical issues as they pertain to environmental problems.

**PROFESSIONAL EXPERIENCE**

- **Detroit, Michigan.** Laboratory Supervisor for a field laboratory established at the Detroit Wastewater Treatment Plant. The project involved a one-year pilot study of the overall operation and plant performance to upgrade and modify existing treatment processes to meet current and future discharge limits. Approximately 20,000 samples were analyzed by seven full-time analysts.
  
- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the development and implementation of an on-site, flow-through bioassay of the plant discharge. The study was performed in conjunction with the Michigan Department of Natural Resources, Water Quality Division.
  
- **Grand Rapids, Michigan. EDI Laboratory Certification.** Direct responsibility for the inorganic parameters analysis and quality control measures necessary for laboratory certification under the Safe Drinking Water Act (SDWA) of 1974. Certification involved both analysis of unknown control samples and corresponding on-site evaluation by the U.S. EPA Region V laboratory certification team.
  
- **Muskegon, Michigan. Uniroyal Chemical Company.** Participated in the soil survey and on-site evaluation of potential soil contamination from deposited chemical waste materials produced by a major chemical company. On-site sample analyses for select parameters were made to locate and detail the extent of contamination.
  
- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the implementation of a treatability study to effectively remove cobalt and samarium from industrial waste. The study results led to the design and installation of treatment facilities.
  
- **Columbia, Missouri. A.B. Chance Corporation.** Responsible for implementing a treatment study for effective removal of heavy metals from process wastewater in order to achieve acceptable discharge limits.



- **Kent County, Michigan. Mill Creek Watershed Management Project.** Participated in the collection, mapping, and interpretation of environmental characteristics to be used as prototype guidelines for the management of area wide streams in the Great Lakes Basin. The project was funded by the Environmental Protection Agency.
- **Three Rivers, Michigan. Hydramatic Division, General Motors Corporation.** Responsible for the analytical services conducted on a survey of process wastewater for an automotive transmission manufacturer. The project involved data collection and analytical services including grab samples, setting automatic samplers on an hourly basis for a seven-day period, and installing recording meters for continuous pH monitoring.
- **Grand Rapids, Michigan. Michigan Department of Public Health Laboratory Certification.** Supervised analytical, bacteriological, and quality control activities involved in achieving certification status for the analysis of potable water supplies in Michigan.
- **Higgins Lake, Michigan. Ralph MacMullan Conference Center.** Served on a three-member panel before a meeting of the Northern Michigan Environmental Health Association. The topic of discussion was an overview of organic chemicals now found in much of Michigan's ground waters. A representative from industry and the MDPH laboratory completed the panel.
- **Grand Rapids, Michigan. Haviland Chemical Company.** Coordinated a static bioassay performed on a water-based detergent utilizing fathead minnows in the 96-hour static test.
- **Sparta, Michigan.** Conducted a dendrological survey of a proposed oil drilling site. The survey was incorporated in an overall environmental assessment of the proposed drilling site.
- **Caledonia, Michigan.** Conducted a dendrological survey of riparian vegetation types located along the banks of the Thornapple River in the area of the Labarge Dam.
- **Grand Haven, Michigan.** Conducted a limnological investigation of the estuary waters of the Grand River watershed near Grand Haven. The collected limnological data were evaluated for potential eutrophication problems resulting from nutrient discharges upstream.
- **Kalamazoo, Michigan. American Cyanamid Company.** Supervised laboratory work required in assisting a major chemical manufacturer with a permit application for existing facility hazardous waste management operation to administratively complete four supplemental technical attachments, multidisciplinary services were required in the areas of hydrogeologic investigation, environmental assessment, failure mode assessment, and engineering review. Field work was completed in 19 days with a report to the client in 25 days to meet scheduled deadlines.
- **Kent County, Michigan.** Coordination of field and laboratory services in conjunction with Act 641 monitoring requirements at two county-owned and operated refuse sites.



Specialized studies were also conducted to identify possible use of landfill gases for electric power generation and the source identification of volatile organic contaminants typical of most municipal landfills.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Implementation of two separate tracer studies aimed at pinpointing possible cracks or defects in the clay liners of four hazardous waste disposal trenches. The study utilized a low absorptivity fluoroscene water soluble dye introduced to each trench. Samples collected from each liner failure detection system were then analyzed for the fluorescent characteristics of the dye.
- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Coordination of field and laboratory services in connection with Michigan Department of Natural Resources Act 64 and U.S. EPA RCRA monitoring requirements. Each sampling event involves collection of ground waters, surface waters, and leak detection monitoring sites.
- **Cascade Township, Michigan. Cascade Resource Recovery/Chemical Waste Management, Inc.** Acted as project chemist and field services coordinator for activities involved in the excavation and site decontamination of an Act 64/RCRA hazardous waste disposal facility. The decontamination program involved the analysis of soils collected in and around each disposal trench after the removal of approximately 20,000 cubic yards of waste materials.
- **Cincinnati, Ohio. Rumpke Waste Systems, Inc.** Acting project manager for a large waste disposal firm headquartered in Ohio, with 20+ landfills located in a 5 state geographical area. Mr. Kriscunas is responsible for coordination of laboratory activities in conjunction with all ground water, surface water, and NPDES monitoring requirements.



**RICK D. WILBURN**

**Quality Assurance Manager**

#### **EDUCATION**

B.S., Environmental Studies, Earlham College, 1985

#### **PROFESSIONAL SUMMARY**

Mr. Wilburn is responsible for all aspects of the laboratory Quality Control/Quality Assurance Program. Primary responsibilities include conducting internal and external auditing of the laboratory, procurement and maintenance of state and federal certifications, and ensuring that all facets of the quality control program remain at the highest level possible. Mr. Wilburn also manages the external and internal Quality Control check sample programs.

#### **PROFESSIONAL EXPERIENCE**

- **TRACE Analytical Laboratories, Inc. – Quality Assurance Manager, 12/95 – 10/96.** Responsible for designing, implementing, and monitoring a formal quality control program. The program included: conducting internal and hosting external audits, implementing corrective actions resulting from any deficiencies, scheduling and reporting performance evaluation sample results, and the review of all Level 5 data packages.
- **EARTH TECH – Organic Laboratory Manager, 10/95 – 12/95.** As Organic Laboratory Manager, Mr. Wilburn was responsible for the day-to-day operations of the organic laboratory, including volatile and semi-volatile analyses by gas chromatography and gas chromatography/mass spectrometry. His responsibilities included scheduling, instrument maintenance, the writing and implementation of standard operating procedures, quality assurance, analytical data review, the technical development of all the organic laboratory personnel, and project management. Mr. Wilburn was also responsible for research and development in the organic laboratory, focusing on ways to automate and improve sample analysis, data quality, and turnaround time.
- **EARTH TECH (Formerly WW Engineering & Science) – Semi-Volatile Laboratory Supervisor, 1/94 – 10/95.** Responsible for the daily operation of the semi-volatile laboratory. The semi-volatile laboratory utilizes gas chromatography, gas chromatography/mass spectrometry, and high performance liquid chromatography in the analysis of semi-volatile organic compounds.
- **WW Engineering & Science – Supervisor, Organic Extraction Laboratory, 4/93 – 1/94.** Supervisor of the staff of chemists responsible for all organic extractions. Accountable for the processing, quality, and turn around of a wide variety of samples involving many extraction techniques and methodologies. Continually experimenting with automation and new technologies to improve extraction quality and turn around time, including solid phase and supercritical fluid extractions.



- **WW Engineering & Science – Supervisor, Mass Spectrometry Laboratory, 9/89 – 1/94.** Supervisor of the staff of chemists analyzing samples for semi-volatile organics in the mass spectrometry laboratory. Oversee all analysis and daily activities involved with the mass spectrometry laboratory. Evaluate, recommend, and implement new technologies. Implementations of these include sub-ambient injections using a Varian SPI injector, sub-ambient temperature programs for optimized chromatography, and the use of ion trap mass spectrometers for lower operating detection limits
  
- **IT Corporation, (formerly PEI Associates, Inc.) – Chemist, Level 3, GC/MS Semi-Volatile Team Leader, 7/88 – 9/89.** Along with daily analysis of samples, responsible for coordinating the efforts of the three analysts and three instruments used for semi-volatile analysis. This included scheduling each instrument/analyst to make sure analyses were completed correctly and on time, training new personnel, instrument maintenance, data checking, and reporting project results to management for client distribution. Leader of GC/MS Quality Circle group.
  
- **PEI Associates, Inc. – Chemist, Level 2, GC/MS Analyst, 12/86 – 7/88.** Primary responsibilities included analyzing soil, water, and other media with an Extrel ELQ-400 mass spectrometer system. Analyses performed included semi-volatile and volatile organics listed on the EPA's Toxic Compounds List according to the Contract Laboratory Program protocol. Also analyzed various other non - Toxic Compounds List compounds using appropriate methods.
  
- **PEI Associates, Inc. – Chemist, Level 1, GC Analyst, 7/85 – 12/86.** Carried out a variety of organic analyses in a wide range of matrixes. Was a primary analyst conducting CLP testing for pesticides and PCBs, and was the primary analyst for routine and non-routine testing for herbicides, and volatile organics.



**JEFFREY P. GLASER**

**Vice President of Laboratory Operations**

#### **EDUCATION**

B.S., Biochemistry, Michigan State University, 1987

#### **PROFESSIONAL SUMMARY**

Mr. Glaser is responsible for the operation and management of the laboratory areas. Main functions include supervision and training of personnel, formulation of standard operating procedures, final approval of laboratory data, and laboratory purchase approval.

#### **PROFESSIONAL EXPERIENCE**

- **TriMatrix Laboratories, Inc., Muskegon – Laboratory Manager, 1994 – 1996.** Responsible for all aspects of laboratory performance. He was responsible for all aspects of laboratory performance including, analytical testing and reporting; business development; customer service; capital expenditures, quality control; quality assurance; laboratory safety; and laboratory profitability. He was responsible for the hiring, training, guidance, and evaluation of all laboratory personnel, and for direction of overall laboratory policies and practices.
  
- **Great Lakes Environmental Laboratories – Senior Chemist, 1992 – 1994.** Mr. Glaser's responsibilities included supervision and training of other laboratory personnel, coordination of sample workloads, data review and evaluation, and quality control. He was also responsible for analysis of pesticides, PCBs, and herbicides using an HP 5890 GC w/ECD detectors.
  
- **Anatech Analytical Laboratories – GC/MS Operator, 1990 – 1992.** Mr. Glaser was responsible for the mass spectrometry analysis of environmental samples in a variety of matrixes for both volatile and semi-volatile organics. For volatiles, Mr. Glaser operated and maintained a Finnigan Ion Trap GC/MS system consisting of a Varian GC and a Tekmar purge and trap autosampler. Primary methodology used was 624/8240. For semi-volatiles, he operated and maintained a Hewlett Packard GC/MSD UNIX-based Chem Station. Primary methodology used was 625/8270. He was also responsible for method development. He served as the Organic Supervisor for the first quarter of 1991.
  
- **Anatech Analytical Laboratories – Volatile Organic Chemist, 1989 – 1990.** Mr. Glaser was responsible for operation and maintenance of two volatile GC systems utilizing ELCD, FID, and PID detectors, and Tekmar and O.I. Analytical purge and trap autosamplers. Primary analyses were 601 and 602.



**STACY K. VANDEN AKKER**

**Human Resources/Business Manager**

**EDUCATION**

B.S. Business Management, Davenport Business College, 1996.

**PROFESSIONAL SUMMARY**

As Business Manager, Ms. Vanden Akker is responsible for the record keeping and review of all financial data for the company. She manages accounts payable, accounts receivable, cash flow, and the generation of financial statements and other management reports. She maintains accurate records for potential audit or other review.

Ms. Vanden Akker also manages all Human Resource functions for TriMatrix Laboratories. She processes payroll on a biweekly basis, coordinates employee benefits, handles internal employee questions and concerns, assures compliance with all federal, state, and local employment laws and regulations, and maintains complete and accurate personnel data files.

**PROFESSIONAL EXPERIENCE**

- **EARTH TECH – Environmental Laboratory Business Office, Administrative Assistant, 9/95 – 1/97.** Responsible for assisting the Business Office Manager with accounts receivable, accounts payable, and the daily input of purchases and invoices.
- **EARTH TECH – Lowell Wastewater Treatment Plant Operator/Laboratory Technician, 8/93 – Present.** Responsible for sample collection, equipment maintenance, and the daily laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates. She is also responsible for the correct input of all results into the reports required by the State of Michigan Department of Environmental Quality.
- **EARTH TECH – Lowell Wastewater Treatment Plant Assistant Laboratory Technician, 8/90 – 8/93.** Assisted the Laboratory Technician in the laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates.



### **3.7 APPROVED SIGNATORIES**

Designated laboratory staff members have the responsibility of validating laboratory documents on behalf of the laboratory organization. General categories and documents requiring a valid signature are presented below.

#### **3.7.1 Client/Invoice Reports**

All laboratory reports compiled and mailed contain at least one representative signature validating the contents of the laboratory report. By default, a report is signed by the appropriate project chemist. Alternate and/or additional signatures include the Laboratory President, Client Services Manager, Technical Director, Quality Assurance Manager, and Vice President of Laboratory Operations. No other individuals are approved to perform signatory approval of client/invoice reports.

#### **3.7.2 Proposals, Price Quotations, and Laboratory Contracts**

Proposals or price quotations for laboratory services contain at least one representative signature, validating the pricing, terms, and conditions of the quotation. At least one representative signature is required. Approved signatures for proposals and price quotations include the Laboratory President, Client Services Manager, project chemists, and a sales or marketing representative.

Required signatures for laboratory contracts are the Laboratory President and a Sales or Marketing representative.

#### **3.7.3 Quality Assurance Project Plans (QAPP)**

Quality Assurance Project Plans contain representative signatures of several responsible parties outside the laboratory. The only laboratory signature generally found on a QAPP is that of the QA Manager. The QA Manager has designated QA/QC responsibilities that are fully documented in QAPP



documents. All QAPPs are signed prior to submission to a governing body or client.

Signatures on the QAPP ensure all procedures, materials, quality control practices and project reports meet the predefined goals of the plan.

#### **3.7.4 Purchase Orders and Agreements**

Because the laboratory spends a significant portion of its annual budget on supplies and equipment, guidelines have been established to document and control purchasing.

Purchasing of general supplies is handled through a contracted vendor within the budgetary guidelines established for each laboratory area.

For major purchases such as equipment, service assessments, or building renovations in excess of \$500.00, purchase orders or agreements must be approved by the Laboratory President or CEO.

#### **3.7.5 Binding Statements - Laboratory Certification Documents or Accreditation**

Many certification or accreditation programs require the laboratory to provide items and statements regarding details on the laboratory's operations and staff. In some cases these statements must be presented to the certifying body accompanied by a binding signature of the laboratory president or CEO.

### **3.8 CAPABILITIES, CERTIFICATIONS, ACCREDITATIONS, AND PROFICIENCY TESTING PROGRAMS**

#### **3.8.1 Capabilities**



TriMatrix conducts analytical laboratory services in support of all major environmental regulations, including CERCLA, RCRA, CWA, CAA, and TSCA.

The laboratory is capable of routinely analyzing a variety of sample matrices, including drinking water, surface water, wastewater, soil, groundwater, solid waste(s), and sludge(s). In addition, analyses have been performed on fish tissue, biota, and air samples by project request.

TriMatrix routinely performs a wide array of environmental and non-environmental, chemical and physical analyses. A list of methods currently utilized by TriMatrix is provided in Appendix B. To maintain a quality system of analytical protocols, TriMatrix uses written Standard Operating Procedures (SOPs) derived from methodology specified by the United States Environmental Protection Agency, other federal and state agencies, and professional compendia.

When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. Unless otherwise specified or required by the client, samples will be subcontracted to a NELAP accredited or ISO-17025 certified laboratory.

### **3.8.2 Laboratory Certification - Federal, State, and Independent**

TriMatrix has been formally recognized for its commitment to quality. The laboratory maintains certification through various federal agencies, as well as several state regulatory agencies and private entities. As required by most of the programs, including NELAP and A2LA, certification and accreditation claims must be made in such a manner as to not imply certification or accreditation beyond that given on the laboratory's actual scope of accreditation. Generic certification or accreditation claims must not be made. The use of symbols (such as the A2LA symbol) and other forms of accreditation must always be analyte and/or method specific. Certification



programs in which TriMatrix currently participates are listed in the subsections below:

**3.8.2.1 Federal Certification/Approval Programs**

U.S. Army Corps of Engineers – DoD QSM

U.S. Air Force – AFCEE

U.S. Army Center for Health Promotion and Preventative Medicine – NELAC/A2LA

U.S. Navy – Navy (IR/QA – DoD QSM)

NELAP – National Environmental Laboratory Accreditation Program

**3.8.2.2 State Certification Programs**

Arkansas	Department of Environmental Quality
Florida	Department of Environmental Protection
Georgia	Environmental Protection Division
Illinois	Environmental Protection Agency
Kansas	Department of Health and Environment
Kentucky	Petroleum Storage Tank Environmental Assurance Fund
Louisiana	Department of Environmental Quality
Michigan	Department of Environmental Quality
Minnesota	Department of Health
New York	Department of Health
Ohio	Ohio VAP Program
Wisconsin	Department of Natural Resources

**3.8.2.3 Independent Certification Programs**

The American Association for Laboratory Accreditation (A2LA)



### **3.8.3 Proficiency Testing Studies**

An integral part of most certification programs are Proficiency Testing (PT) Studies. PT studies are analyzed periodically as external “blind” or “double blind” spiked samples containing specific (known only to the administrators of the study) concentrations of target analytes. The laboratory reports the results to the agency or firm administering the PT study. The administrator then evaluates the laboratory’s performance based on a comparison of the reported values with the known analyte concentrations. Laboratory results are scored and reports are prepared by the study administrator. The reports are submitted to the laboratory, certifying programs, and agencies or private entities that subscribe to the program.

TriMatrix routinely participates in the following proficiency testing programs:

- Water Supply (WS) Study
- Water Pollution (WP) Study
- Soil PT Study
- USEPA DMRQA

## **3.9 LABORATORY FACILITIES, EQUIPMENT, AND SUPPLIES**

### **3.9.1 Physical Plant**

#### **3.9.1.1 Laboratory Demographics**

The current TriMatrix Laboratories facility, located at 5560 Corporate Exchange Court SE, Grand Rapids, Michigan, was constructed in 1999. The 20,000 square foot structure was designed predominantly by the laboratory staff, with careful consideration given to the strict analytical testing requirements of today’s environmental marketplace. Special attention was given to the sample preparation areas and the segregation of non-



compatible areas such as semi-volatile and volatile organics. Samples are stored according to type, with a large centrally located walk-in cooler used for the storage of all non-volatile, non-hazardous waste samples, to which both the sample receiving personnel and the laboratory staff have ready access. Quiet office areas were also built in, to provide space for data review, report compilation, and technical review discussions. A breakdown of each general area of analysis and the space allocated is as follows:

<b>Laboratory Area</b>	<b>Space Allotted, ft<sup>2</sup></b>
Wet Chemistry/Microbiology	Approx. 2000
Atomic Absorption/Emission	Approx. 2000
Volatile Organics	Approx. 1600
Semi-Volatile Organics	Approx. 2300
Sample Processing & Storage	Approx. 2400
Administrative Offices	Approx. 4200
Organic Pretreatment	Approx. 1300
Miscellaneous Space	Approx. 4200

The attached facility layout (Figure 3-6) shows the general lab areas and other space allocations.

Access to all laboratory areas including sample storage, sample container preparation, sample preparation, sample disposal, documents storage and clients files are secured. Non-authorized personnel may enter these areas only if escorted by a laboratory staff member.

Project initiation, sample control, and analysis, are all controlled using a Laboratory Information Management System (LIMS).

Under the direction of the Laboratory President, TriMatrix is organized into the following operating areas and support services.



### **Laboratory Administration**

Client Services  
Data Management  
Sales/Marketing  
Project Management  
Health and Safety  
Quality Assurance  
Computer Services

### **Analytical Operations**

Inorganic Laboratory  
    Metals Laboratory  
    Non-Metals Laboratory  
Organic Laboratory  
    Volatile Organic Laboratory  
    Semi-Volatile Organic Laboratory  
    Organic Extraction Laboratory

(Refer to Figure 3-2 for a graphical representation of the Laboratory Organization Chart)

#### **3.9.1.2 Reagent Water Systems**

Laboratory water originates from the Grand Rapids potable water distribution system. At the laboratory, the water is softened and passed through an activated carbon filter to remove residual chlorine. The water then enters a reverse osmosis system where approximately 90% of the dissolved constituents are removed. The water is temporarily stored in a 120 gallon holding tank until demand activates a mechanical pump that transfers the water through two mixed bed deionizing canisters. This water meets the requirements of ASTM Type II, and is utilized for glassware cleaning and as a feed-water to a variety of polishing systems.



The polishing systems are comprised of a distillation unit and a Milli-Q 4 Bowl System. Distilled RO-Deionized water is used primarily for BOD and metals analyses. Mill-Q water, which is equivalent to an ASTM Type I designation, is primarily used for the preparation of standard solutions and reagents.

Each water system is periodically monitored for specific quality requirements. Monthly, heterotrophic plate count and total residual chlorine analyses are performed. Weekly, the water system itself is checked for operational readiness and a hardness test is performed. Daily, additional readiness checks including a conductivity test are performed.

Responsibility for monitoring the TriMatrix reagent water systems is carried out by the Quality Assurance Department and personnel in the inorganic wet chemistry laboratory.

#### **3.9.1.3 Ventilation Systems**

The laboratory ventilation system was specifically designed to minimize or eliminate airborne contamination. Externally, the air conditioning unit intakes were located taking into consideration prevailing wind patterns, positioning them upwind of the fume hood exhaust stacks. Taking into account wind-shifts, the exhaust stacks were equipped with high velocity fans to disperse potential contaminants well above the building. Internally, the air-handling systems controlling heating, cooling, and humidity, also maintain maximum cfm air turnover. Additionally, the air-handling systems are monitored and controlled via a NOVAR computer controller.

#### **3.9.1.4 Compressed Air**

Compressed air must be free of dirt, water, and oil. Compressed air purchased from vendors is high purity grade (breathing air).



Compressed air produced in the laboratory uses filters at the compressor to remove water from the delivery lines. For the gas chromatographs and atomic absorption spectrophotometers, additional filters are located on the instrument to remove any residual oil at the point of use.

#### **3.9.1.5 Electrical Services**

The electrical system in use at TriMatrix was designed specifically for a laboratory environment. Special attention was paid to instrument requirements, including the isolation of separate lines for critical applications like GC, GC/MS, atomic absorption, and automated analyzers.

All laboratory benches, hoods, and work areas were designed with sufficient outlets to accommodate a variety of laboratory applications, such as distillations, digestions, and extractions.

Surge protection devices are in place for all laboratory computing equipment. The laboratory LIMS system is also protected by an Uninterrupted Power Supply (UPS). This UPS allows for a sequenced shutdown of the LIMS system during a power failure. This sequenced shutdown provides excellent protection of the LIMS database during a power interruption.

#### **3.9.2 Equipment, Supplies, and Chemical Procurement; Reception, Storage, and Inventory**

For an environmental testing laboratory where trace analyses are routinely performed, certain specifications for laboratory equipment, supplies, and chemicals are critical to quality. A minimum specification for accuracy and precision of equipment such as analytical instrumentation, balances, glassware, and water baths is required for each analytical procedure. The Technical Director in conjunction with the Laboratory President and laboratory area



managers are responsible for determining minimum specifications before equipment is procured. The analytical specifications are based on a detailed review of the test methods. Purchasing is coordinated through the purchasing department. Records are maintained on all vendors exhibiting poor performance on either their service or product. Relationships will be terminated with any vendor whose records indicate sub-standard performance.

#### **3.9.2.1      Equipment Management/Maintenance/Inventory**

A sufficient inventory of equipment is maintained to prevent testing delays resulting from equipment failure. Service is performed on equipment on a scheduled basis. A stock supply of spare parts that are known to wear out regularly is maintained.

Adequacy of equipment for its intended purpose must be verified before use. Maintenance logbooks are kept to document maintenance procedures on major equipment, allowing preventive maintenance frequency and requirements to be determined. Maintenance procedures are discussed in the various analytical SOPs.

A complete listing of Laboratory Equipment is presented in Appendix C of this manual.

#### **3.9.2.2      Glassware**

Only glassware providing the required precision is used for a particular analytical procedure. TriMatrix purchases Class A pipets, burettes, and volumetric flasks, to meet this specification. A standard operating procedure is utilized for cleaning each type of glassware. Cleaning of glassware is performed according to the analysis being conducted and the sample matrix involved, but certain general rules apply to all glassware washing procedures:



- Use hot water to wash away water-soluble substances.
- Use detergent, dichromate solution, organic solvent, nitric acid, or aqua regia to remove other materials according to the specific glassware cleaning procedures.
- Avoid using detergents on glassware to be used for phosphate determinations.
- Use ammonia-free water for ammonia and kjeldahl nitrogen analyses.

For all analyses, it is advisable to rinse glassware with tap water followed by deionized water immediately after use, as residue allowed to dry on glassware is more difficult to remove.

### **3.9.2.3 Reagents, Solvents, and Gases**

Purchasing of reagents, solvents, and gases are carefully controlled through an ordering system that maintains a minimum level of quality in the testing process. The Quality Assurance Department defines the suitable grades of ordered materials. Designates from each laboratory area verify upon receipt that incoming materials meet these requirements. Certificates of Analysis are forwarded to the Quality Assurance department where they are scanned and stored. Each laboratory area will monitor the proper storage and the eventual removal of reagents, solvents, and gases, when their shelf life has expired. All consumable reagents and chemicals must be labeled with the date received to ensure a First-In-First-Out (FIFO) system of use.

Reagents, solvents, and gases are available from vendors in a broad range of purity, from technical to ultra pure grades. The analysis, as well as the sensitivity and specificity of the method, must be considered when choosing a grade. Analytical reagent (AR) grade is suitable for most inorganic analyses. Trace organic analyses frequently require ultra pure grades. AR grade is the minimum



approved for reagents used in organic analysis. The absence of certain impurities is required for some GC detectors - notably sulfur and phosphorus in an FID detector. Trace metals analyses including atomic emission and atomic absorption spectroscopy usually requires spectro-quality reagents, although AR grade may be suitable in some cases. Florisil, silica gel, and alumina used as absorbents in organic extract cleanups, must be checked for interfering components and activated according to the analytical method. Compressed gases are available in various purities, usually expressed as a percent (e.g. 99.999). Gases are filtered in the laboratory delivery lines to remove moisture, oil, and other contaminants. Refer to the analytical method and instrument manufacturers operating manual for gas purity requirements.

Provided they are available, expiration dates of unopened chemicals are based on the date determined by the manufacturer. They may also be derived from the analytical method. A new expiration date may be required once the chemical is opened. The following guidelines are utilized in assigning expiration dates:

**Unopened Reagents, Solvents, and Neat Chemicals**

Manufacturers assigned expiration date or 5 years from date received, whichever occurs first.

**Opened Reagents, Solvents, and Neat Chemicals**

2 years from date opened or remainder of manufacturers assigned expiration date, whichever occurs first.

**Prepared Solutions - Stock**

Manufacturers assigned expiration date or 1 year from date opened, whichever occurs first.

**Prepared Solutions - Working**



Assigned expiration date of stock, or 6 months from date prepared, whichever occurs first.

Unpreserved ethers have an expiration date of 34 days due to the potential for peroxide formation.

In order to maintain expiration date accuracy it is critical that the date opened is recorded on all containers, and the expiration date originally entered into the LIMS system be updated based on the date opened.

#### **3.9.2.4 Certified Standards**

The purity and traceability of standards used in the analytical process is crucial to the quality of the data generated. Only high quality standards certified by established vendors are to be utilized. Calibration standards must be of the purity required by the method for a particular analysis.

Upon receipt all purchased standards are entered into the LIMS system and labeled with a unique identifier and an expiration date. The date received is also recorded on the container. Stock and working standards are likewise labeled.

All calibration standards are validated against a second source standard. A second source standard is analyzed with every initial calibration. The quantitated value is compared to laboratory established limits. Recovery must fall within these limits for the calibration and calibration standard to be considered acceptable. Stock and working standards are also monitored for visible signs of deterioration (precipitates, color change, volume change).

Vendor expiration dates for purchased stock standards must not be exceeded. Expiration dates for laboratory prepared standards are



based on guidelines in the analytical method, generally 6 months for working, and 1 year for stock standards.

### **3.9.2.5 Chemical / Reagent Storage**

Bulk chemicals and reagents are stored in a several locations and under a wide variety of conditions within the laboratory. Specific storage conditions for many reagents are presented in each laboratory testing SOP. Additional storage information is referenced in both the TriMatrix Laboratory Safety Manual and the TriMatrix Chemical Hygiene Plan. For general purposes, the following storage conditions are used:

<b>Chemical /Reagent Type</b>	<b>General Storage Requirements</b>	<b>Location/Lab Area</b>
1) Bulk Dry Chemicals	Dry Chemical Storage Cabinets	Inorganic Laboratory
2) Inorganic Acids	Vented Acid Storage Cabinets	Metals Laboratory
3) Organic Solvents-Flammable	Vented Flammable Cabinets	Inorganic & Prep Laboratory
4) Organic Solvents-Nonflammable	Vented Storage Cabinets	Inorganic & Prep Laboratory
5) Compressed Gases	Secured Gas Storage Area	Garage & Outside Storage
6) Bacteriological Materials	Reagent Refrigerator	Inorganic Laboratory
7) Aqueous Standards	Reagent Refrigerators	All Laboratory Areas
8) Organic Standards-Flammable	Explosion Proof Refrigerators and Freezers	Organic Laboratory Areas
9) Organic Standards-Nonflammable	Standards Refrigerator & Freezers	Organic Laboratory Areas
10) Sample Extracts	Extract Freezers	Organic Laboratory Areas
11) Digestates-Metals	Vented Acid Storage Cabinets	Metals Laboratory

## **3.10 TRAINING**

Proper training of laboratory personnel is an essential part of staff development. Training procedures include documentation of training activities completed and serve as a guideline for continual staff development. All testing personnel must familiarize



themselves with the laboratory's training procedure (TriMatrix SOP GR-10-109) and implement all associated policies and procedures.

Personnel files contain the training documentation related to the development of each laboratory employee. Included are in-house training, external training certificates, safety training, ethics training, and other materials specific to the analyst. The quality assurance department maintains the training file system.

### **3.10.1 Training Orientation**

The human resources department initiates training orientation for each new employee on the first day of employment. Orientation includes completion of various training checklists (Appendix D). These checklists provide documentation of the orientation after being signed by the new analyst and the trainer and become a part of the employee's permanent training record.

### **3.10.2 Code of Ethics/Data Integrity Training**

It is the intent of TriMatrix Laboratories, Inc. to consistently report data of the highest quality. For this to be possible, analysts are instructed in accordance with the level of data quality desired and are provided with an environment conducive to its achievement. Besides providing the analyst with all necessary supplies and equipment, the work environment is maintained as free from undue pressures as possible. Such pressures may be through internal peer pressure or deadlines, or through external customer complaints or priority requests. It is the responsibility of management to insulate the analyst from such pressures as much as possible. Data quality cannot be compromised without reason and the analyst will not be reprimanded for adhering to established quality protocols in the face of such pressures.

During the orientation with human resources, these policies will be explained and the employee asked to review and sign a Code of Ethics/Data Integrity Policy Agreement (Appendix E). This agreement documents the understanding between management and the new employee concerning management's



position on data quality, sample analysis and data reporting, and the consequences of improper actions. The signed agreement is retained as part of the employee's permanent record.

### **3.10.3 Document Storage**

All essential laboratory documents are stored on the laboratory's intranet drive. During orientation, the new employee is shown how to access these documents and instructed on which ones are required reading. These include the Quality Assurance Manual, Chemical Hygiene Plan, Safety Manual, Employee Handbook, a memo containing instructions on TriMatrix error correction policies and standard operating procedures. Forms are signed documenting that the employee has read and understood these documents.

### **3.10.4 Demonstrations of Capability (DoC, IDC, CDC)**

All analysts and instruments used for sample analysis must complete at least one type of Demonstration of Capability (DoC). Three types of demonstrations exist: a method/instrument DoC, an analyst Initial Demonstration of Capability (IDC) and an analyst Continuing Demonstration of Capability (CDC). All demonstrations of capability are documented, reviewed, and signed in accordance with the TriMatrix SOP for analyst training (GR-10-109). All supporting data necessary to reproduce the DoC, IDC, or CDC must be available. Sample analysis may not begin without the successful completion of an appropriate DoC and submission of all associated paperwork to the Quality Assurance Department.

#### **3.10.4.1 Demonstrations of Method Capability**

Prior to the acceptance and institution of any Standard Operating Procedure or the use of any new instrument, a satisfactory demonstration of method/instrument capability study is required. This DoC must be performed on all instruments used for the analysis. This is a one-time study, unless there is a significant



change in the instrument or methodology. This procedure must be successfully completed for all applicable matrices prior to sample analysis. The instrument DoC consists of a Demonstration of Accuracy and a Method Detection Limit Study (MDL); two separate studies demonstrating the instrument's capability of producing sufficiently accurate and sensitive results.

### **1) Demonstrations of Instrument Capability**

For instruments that utilize an initial calibration, an acceptable initial calibration will serve as the demonstration of capability. The low point of the calibration must be at or below the lowest desired reporting limit. The high point defines the calibration range. Any sample with an analyte concentration above the high point in the calibration requires a dilution before quantitation. For procedures not using a calibration curve, seven standards at various concentrations covering the range of the analysis must be analyzed to demonstrate accuracy throughout the range. These standards must be prepared from the same source as that used for calibration. The relative standard deviation of the average recovery must be less than 20% and average percent recovery must be between 95 - 105%. The spreadsheet in Appendix F and the form in Appendix G must be completed to document the accuracy test. Return these completed forms to the Quality Assurance Department.

### **2) Method Detection Limit study (MDL)**

A Method Detection Limit (MDL) study is performed in accordance with TriMatrix SOP GR-10-125. MDL studies must be completed for each matrix-specific preparative and/or analytical technique and must be updated annually or whenever a major change is made to the preparative and/or



analytical technique. The MDL procedure is described in section 3.11.2.

#### **3.10.4.2 Initial Demonstrations of Analyst Capability**

After orientation and training, each analyst must complete a successful IDC study. The IDC, unlike the DoC, is not instrument dependent. An IDC must be completed any time a significant change to a procedure occurs. Conduct the IDC study by preparing four replicate blank spikes (for any procedure with a pre-treatment) or four replicate second-source calibration verifications (for any procedure without a pre-treatment) at a concentration in the lower half of the calibration or analytical range. In either case, the spiking standard must be prepared from a source other than that used for calibration. For analyses where a spiking standard is not an option, the acceptable analysis of a single blind PT sample will suffice. Alternatively, the analyst may analyze four replicates of a client sample against four replicates of the same sample analyzed by an experienced analyst for statistical comparison.

Process the four spikes, PT sample, or replicates, following every step in the preparative and/or analytical procedure concurrently or over a period of no more than 72 hours. Enter all four results into the IDC spreadsheet (Appendix H) or all eight replicates into the IDC spreadsheet (Appendix I) as appropriate. The spreadsheet will calculate average percent recovery and relative standard deviation then evaluate against default acceptance criteria (which may need changed to fit the procedure). If all acceptance criteria pass, the analysis of actual samples may begin.

When one or more analytes fail any criterion, the study is unacceptable for the failed analyte. Locate and correct the source of the problem then repeat the study for the failing analyte successfully. If none of the options presented above are possible



(such as with the TCLP pre-treatment), the analyst must perform and submit an acceptable method blank with acceptance being that all analytes are at or below the method detection limit.

When complete, forward the IDC spreadsheet, the NELAC Demonstration of Capability Certification Statement (Appendix J), the Laboratory Training Checklist (Appendix K), the MDL study when necessary (Appendix L), and/or PT results to the Quality Assurance department for review and training documentation.

#### **3.10.4.3 Continuing Demonstrations of Analyst Capability**

A Continuing Demonstration of Capability (CDC) is required annually. In addition to the IDC study described in section 3.10.4.2, the CDC may be accomplished by inputting the last four results of an MDL study to the IDC spreadsheet if completed exclusively by the analyst, by inputting four *consecutive* SCV results obtained during the course of routine sample analysis if completed exclusively by the analyst or by exclusively running a PT study analysis successfully.

When complete, forward a copy of all applicable data necessary to reconstruct and validate the study to the Quality Assurance department for training documentation.

#### **3.10.4.4 SOP Revision Checklist**

SOPs are periodically reviewed and updated. When an update is released, the appropriate form from Appendix M must be completed to record that the applicable analysts have read, understood and agree to follow the revised SOP.

### **3.10.5 Continuing Training and Education**



TriMatrix Laboratories, Inc. is committed to education and training on a continual basis for employees. There are various ways in which continuing education may occur, including:

- seminars
- cross-training for additional job responsibilities
- retraining
- method and technology updates

### **3.11 DETECTION LIMITS**

The process of quantifying an analyte in an environmental matrix using specific analytical procedures must use detection limits as a point of reference. The three levels of analytical detection are described below.

#### **3.11.1 Instrument Detection Limit - IDL**

Most analytical instruments produce a signal even when a blank (matrix without analyte) is analyzed. This signal is referred to as the noise level. The IDL is the analyte concentration required to produce a signal greater than three times the standard deviation of the instrument noise level. The IDL can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. If the instrument does not give a signal for the blank, perform the study using standards at the expected IDL concentration. Each measurement should be performed as though it were a separate analytical sample followed by a rinse and/or any other analytical step normally performed between the analysis of separate samples. Where required by the method (for example, SW-846 method 6010B), IDLs need determined at least every three months or at a project-specific designated frequency and kept with the instrument.



The IDL only defines an instrument's limitations and does not take into consideration sample processing in preparation for the analysis. As such, it may not be used to estimate the method detection limit (Figure 3-7).

An IDL study is required only when specified by the analytical method reference.

The requirement for performing an IDL study does not negate the requirement of a method detection limit (MDL) study.

### **3.11.2 Method Detection Limit - MDL**

Many times there is more to an analysis than a direct analysis such as a digestion, dilution, concentration, chemical treatment, extraction, and cleanup. Additional analysis steps propagate error from the uncertainty associated with each step. Since the method detection limit is defined in terms of error, all steps leading to analysis must be included to calculate the MDL (Figure 3-7).

The MDL is defined as the minimum concentration of a substance that can be detected and reported with 99 percent confidence (statistically) that the value is above zero. The MDL is calculated from spiked blanks which go through the entire sample preparation and analysis scheme. MDL studies are run for aqueous and solid methodologies for every analyte targeted. Although MDL studies are completed for all laboratory determinations, if any result were to be obtained without an associated MDL study, it must be reported as estimated. All calculated MDL values must be verified.

The MDL procedure used at TriMatrix Laboratories references 40 Code of Federal Regulations, Part 136, Appendix B where seven replicate aliquots of laboratory reagent water (for an aqueous methodology) are spiked with every analyte of interest at the estimated minimum practical quantitation limit (PQL). For a solid methodology, an inert substance or empty vessel is spiked. The PQL may be estimated using instrument noise, a series of method blanks, the



instrument calibration, or the preliminary MDL estimation as described in TriMatrix SOP GR-10-125.

It is essential that all sample preparative, cleanup, and analytical steps be included in the MDL study. Calculate MDL study results based on all computations required to achieve the final result in sample-designated units.

To calculate the MDL, input all seven results to the MDL spreadsheet located on the laboratory intranet library. The spreadsheet calculates the MDL by multiplying the standard deviation by 3.143 which is the one-sided t-distribution for seven samples (with six degrees of freedom) for a 99% confidence interval. There must be no zero percent recoveries in the dataset and the concentration spiked must be between 1 and 5 times the MDL value.

Repeat the study at a lower concentration if results in the MDL spreadsheet are flagged “Fails Too Good”. Repeat at a higher concentration if the MDL value is flagged “FAIL”. However, if the spiking concentration is chosen based on a good estimate of the actual MDL, the MDL study should not fail. Re-estimate the actual MDL based on the failed MDL value before repeating the study.

**Note:** Even if the MDL value appears acceptable, the MDL procedure is not complete until an MDL verification also has been successfully performed.

The MDL verification is accomplished by analysis of a method blank and blank spike. Prepare the blank spike at a concentration between 1 – 4 times the calculated MDL value. If the blank spike response is greater than or equal to three times that found in the method blank, the MDL verification passes and the calculated MDL value is acceptable.

If the blank spike response is less than three times that found in the blank, the MDL value is too low. Repeat the MDL study by estimating the concentration necessary to produce a response equal to or greater than three times the method blank and repeat the verification study. Repeat the MDL verification. Repeat



until the MDL verification is at least three times the method blank. Only the MDL value or MDL verification that passes the MDL verification criterion may be used as the calculated MDL.

Appendix L shows an example of the MDL spreadsheet used to calculate and verify MDL values and practical quantitation limits.

The MDL for all analytes in aqueous and solid methodologies must be determined annually or whenever a significant modification is made to the procedure.

### **3.11.3 Minimum Practical Quantitation Limit - PQL**

The PQL is defined as the minimum concentration of an analyte that can be quantitatively reported (versus qualitatively detected) within specified precision and accuracy limits under normal laboratory operating conditions. (Figure 3-7).

The minimum PQL is the analyte concentration spiked in the MDL study or 3 times the concentration spiked in the MDL verification when the initial MDL value fails. The minimum PQL must be 3-10 times the MDL value.

Note: Practical quantitation limits actually achieved for any given sample analysis will be highly dependent on the matrix and/or required dilutions.

## **3.12 PROCEDURES FOR ACCEPTING NEW WORK/TESTS**

### **3.12.1 New Test Requests, Development, and Approval**

Client Services must submit a request for new analyses to each impacted laboratory area where the request will be formally processed. Evaluation of the request will include the suitability of the analyte for quantitation, availability of existing test methods, instrumentation, capacity, standard materials, etc. The

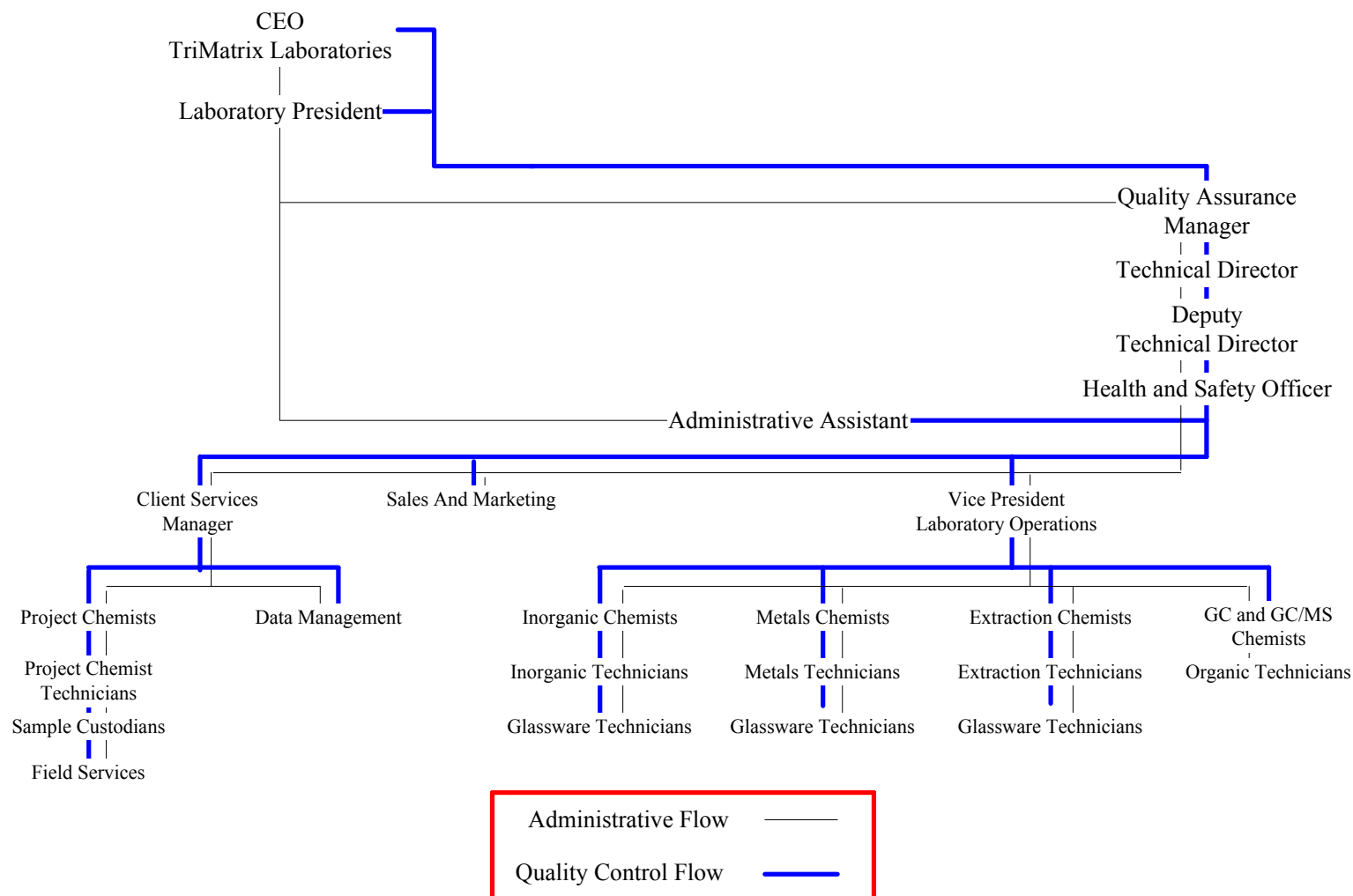


Vice President of Laboratory Operations, Technical Director, and/or Group Leader will provide a prompt response to client services to ensure client needs can be addressed.

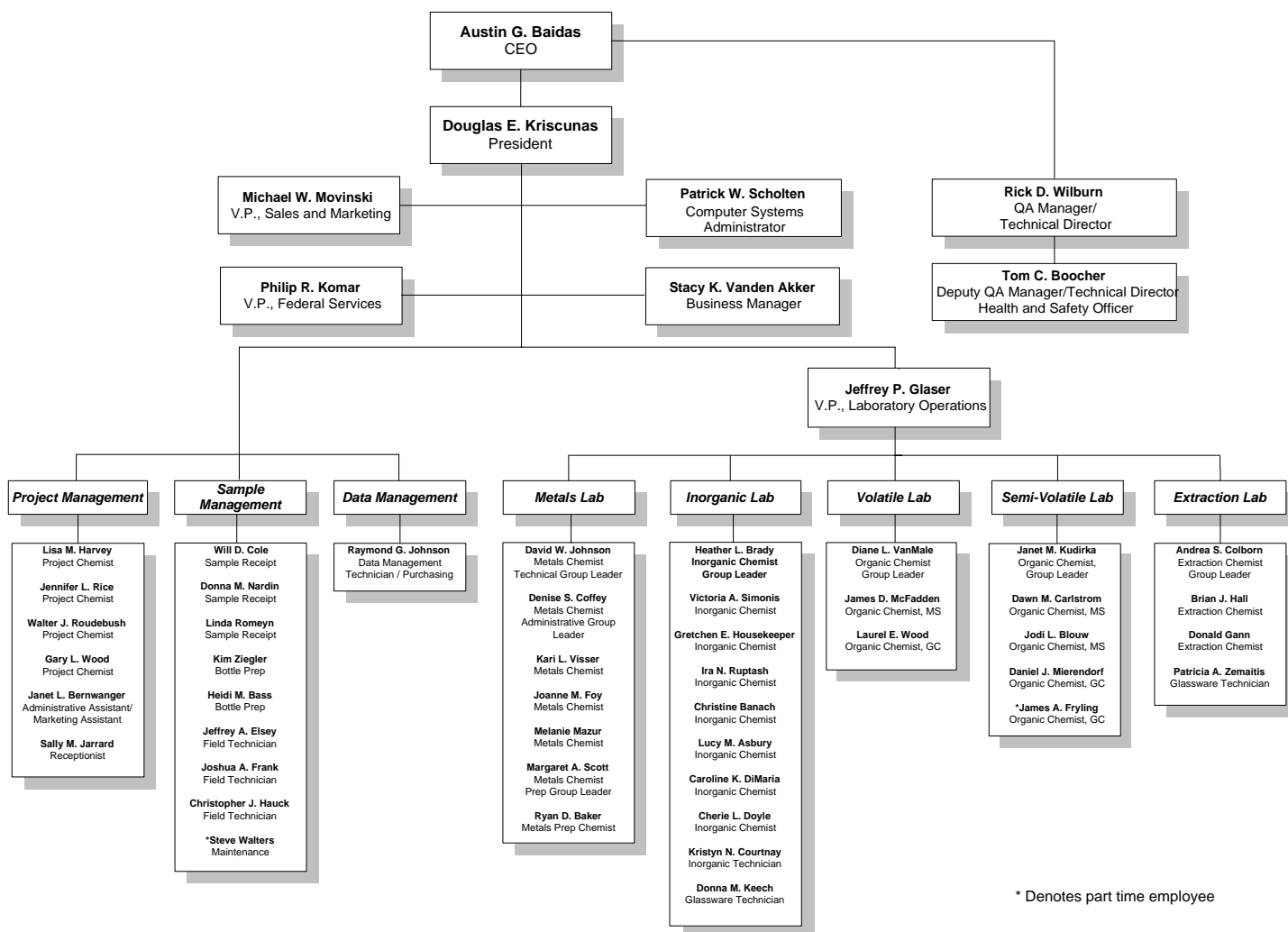
All newly developed procedures are reviewed by the laboratory Technical Director and must comply with all requirements outlined in section 3.10.4.



**Figure 3-1**  
**Quality Control Chain of Command Flow Chart**

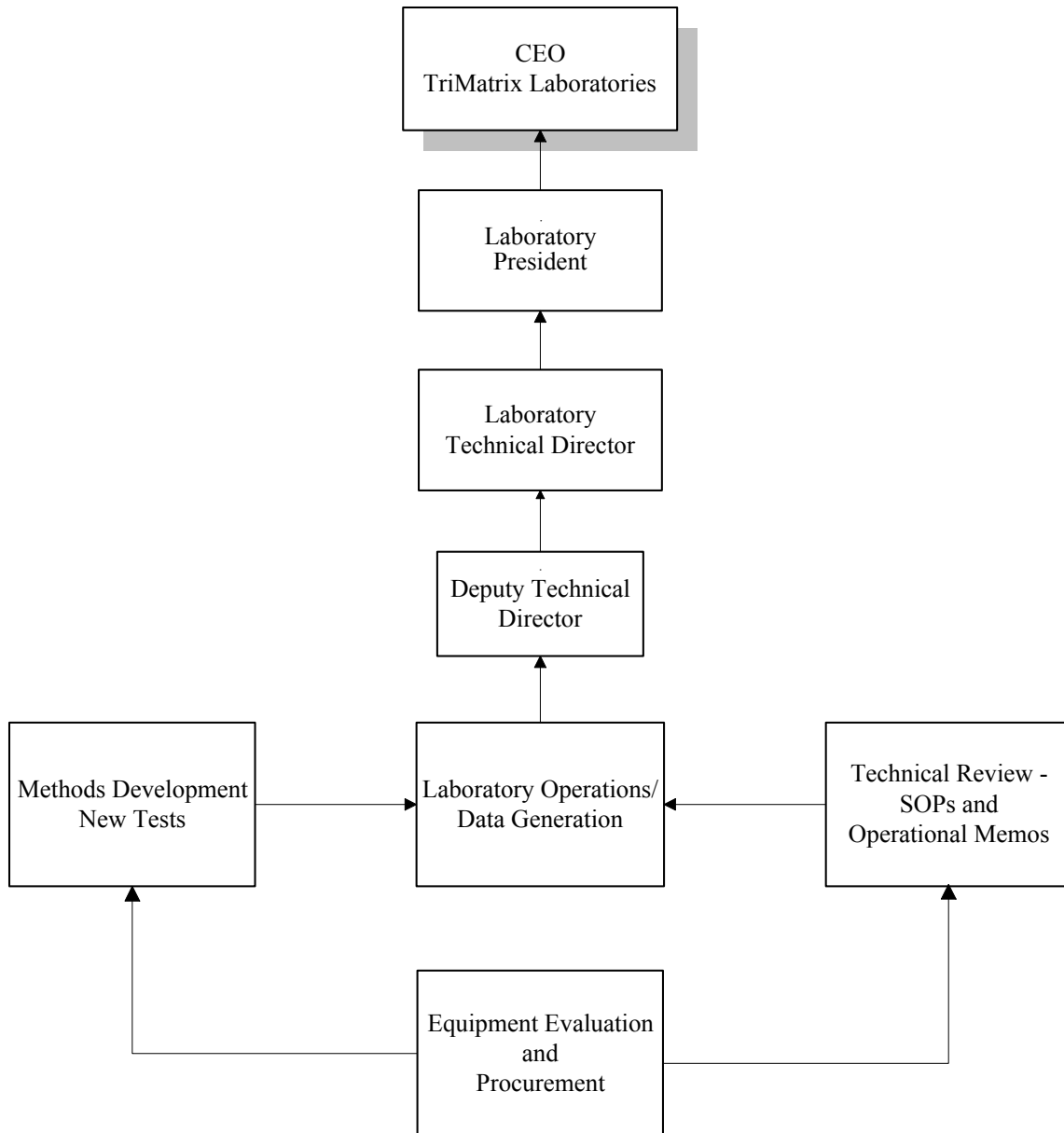






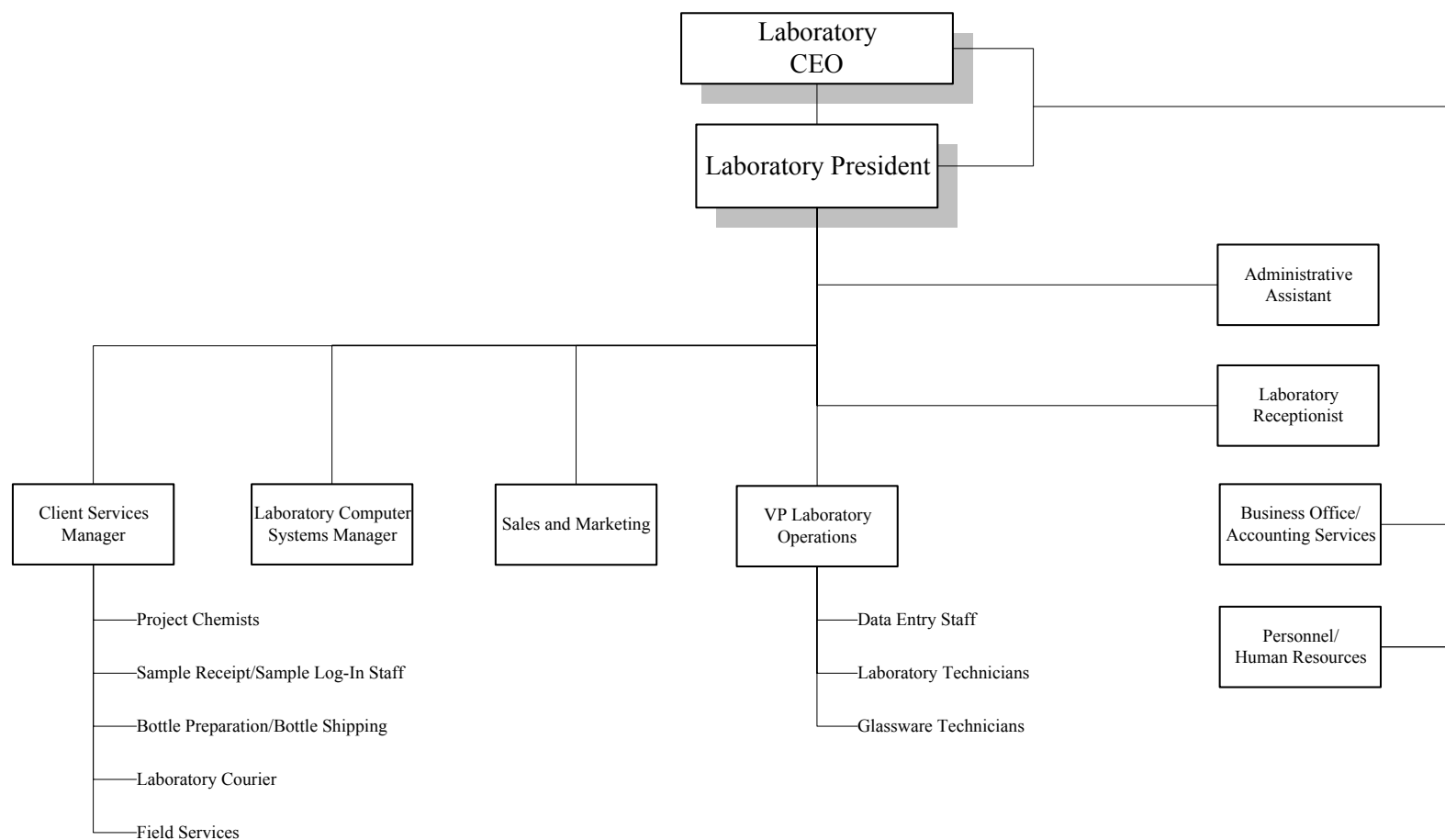


**Figure 3-3**  
**RELATIONSHIPS**  
**Management to Technical Services**



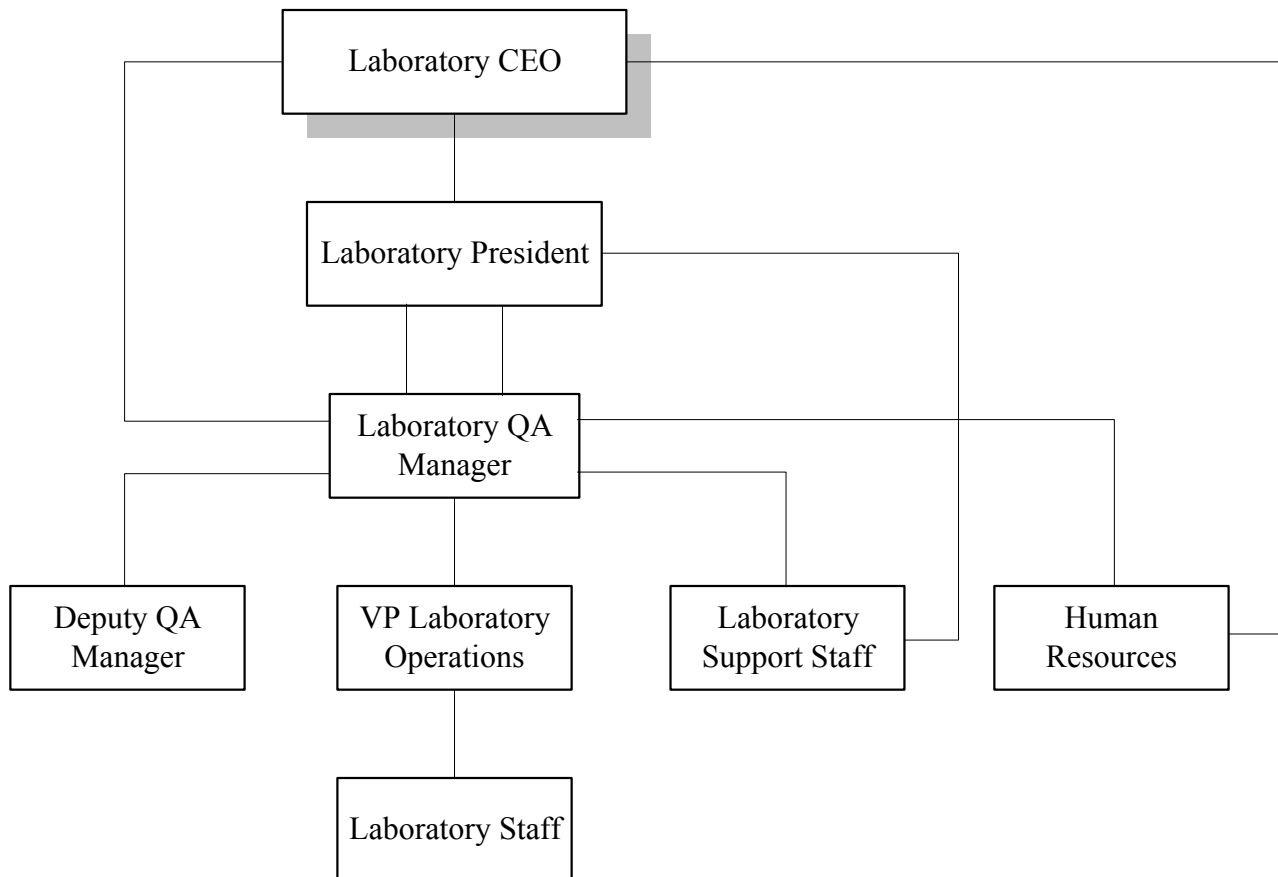


**Figure 3-4**  
**RELATIONSHIPS**  
**Management to Support Services**





**Figure 3-5**  
**RELATIONSHIPS**  
**Management to Quality System**

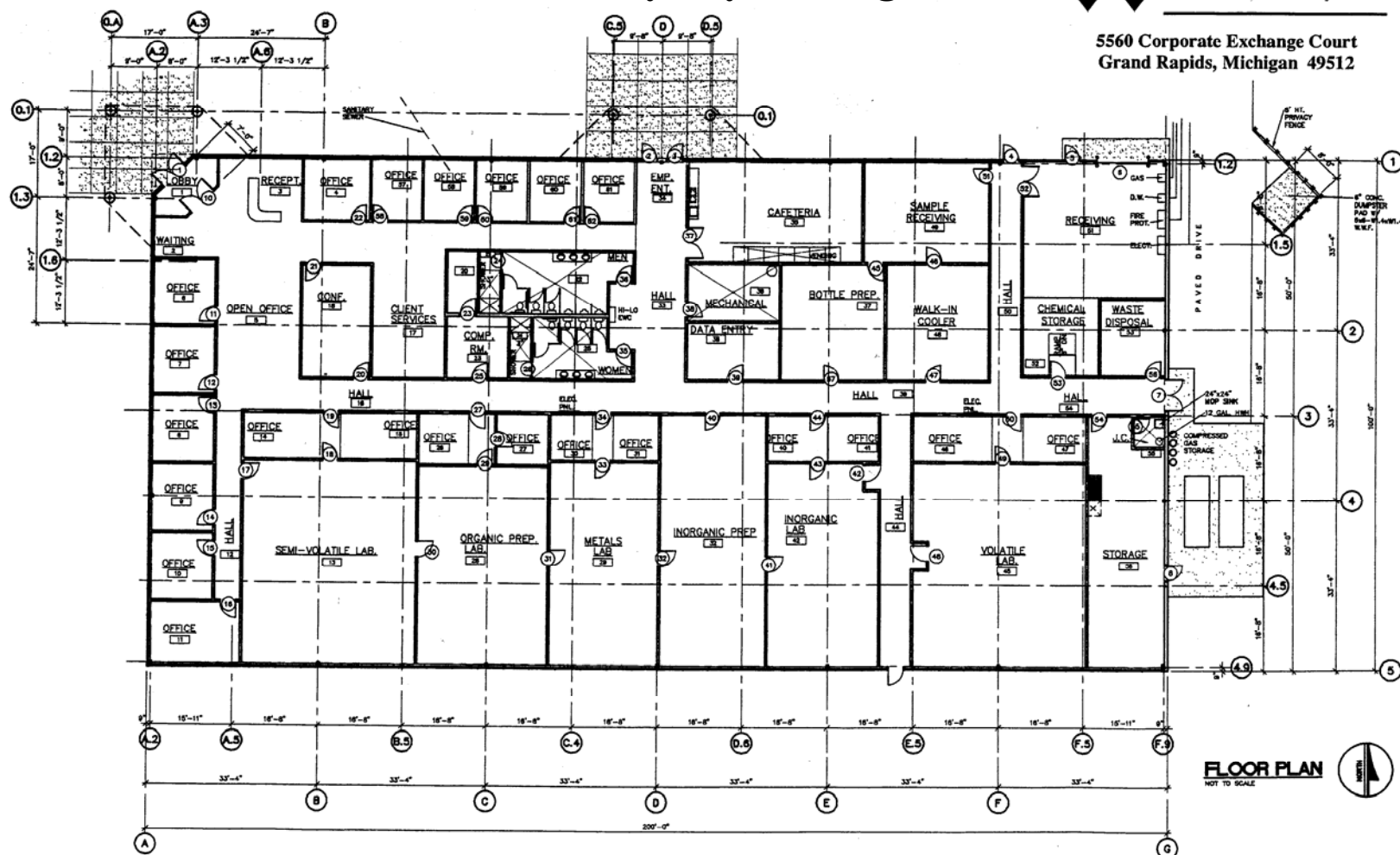




**Figure 3-6**  
**Laboratory Layout/Diagram**

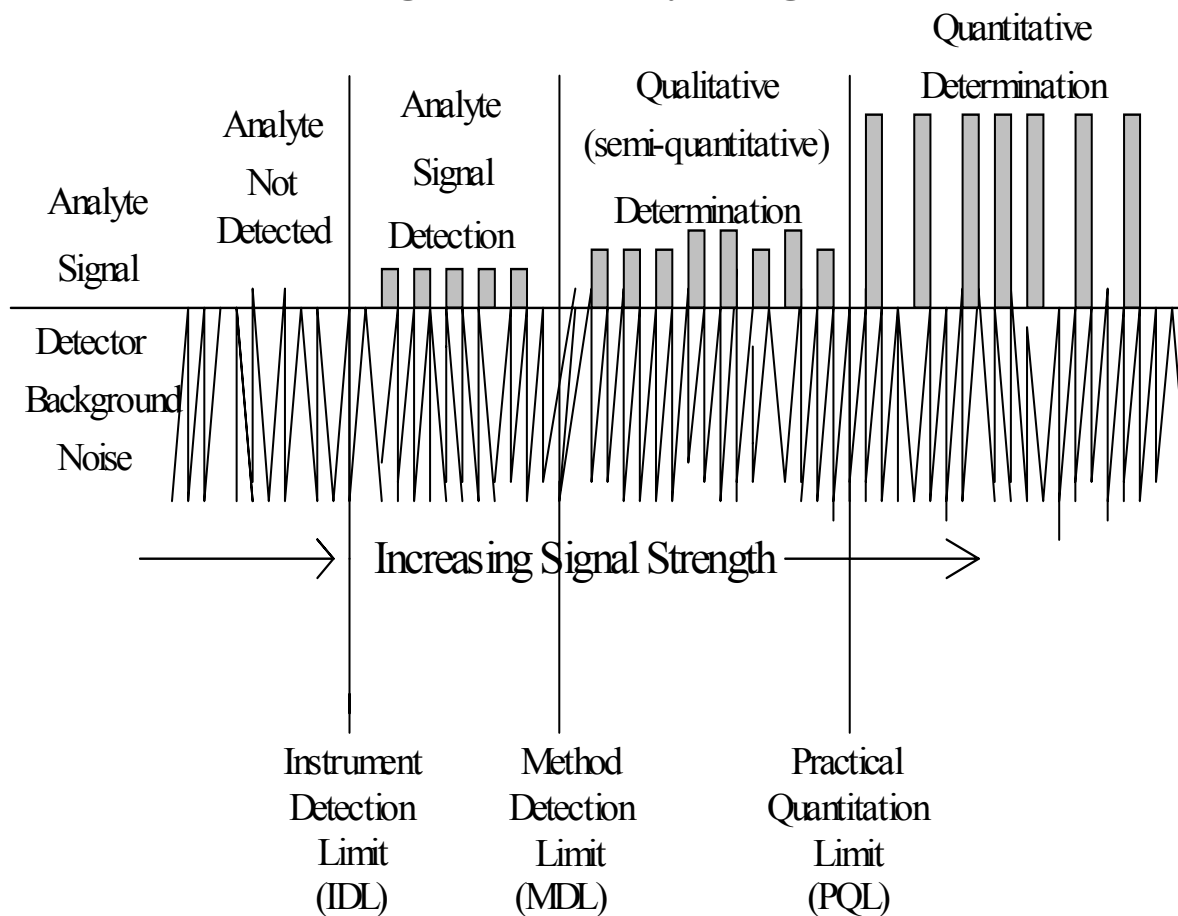


5560 Corporate Exchange Court  
 Grand Rapids, Michigan 49512





**Figure 3-7**  
**Regions of Analyte Signal**





## **4.0 QUALITY CONTROL**

### **4.1 DOCUMENT CONTROL AND MAINTENANCE**

#### **4.1.1 Procedures for the Control and Maintenance of Documentation**

Documents utilized in the quality system are subject to strict control regarding their creation, revision, approval, use, and distribution. This applies to documents generated both internally, and those received from outside sources. Obsolete documents that are retained in circulation for either legal or knowledge preservation purposes are marked as “obsolete”. The structure of the documentation used in the TriMatrix quality system is presented in Figure 4-1.

##### **4.1.1.1 Internal Documentation**

Examples of internal documentation include Standard Operating Procedures, the Quality Assurance Manual, miscellaneous forms, and logbooks. All documents must be reviewed and approved by one or more senior staff prior to their use. All documents will print with both the file name and revision number. Where possible, the document will contain the TriMatrix logo. All logbooks must be bound and paginated.

All approved documents are stored on the laboratory intranet read only drive designated as “Library.” Document control is maintained through the use of the laboratory computer network. By maintaining only the current version of an approved document on the Library drive, document control and security is maintained. This procedure provides immediate access to the latest revision of all documents.

Document revisions may be made by any applicable, qualified, laboratory employee. Minor document revisions, such as those required to a Standard Operating Procedure, may be made by hand.



All hand amendments must be legible, dated and initialed, and recorded in ink. All hand amendments must be approved by, and distributed through, the Quality Assurance Department. Hand amendments will be incorporated into the next revision of the document. Hand amendments cannot be used for major document revisions. Extensive revisions require a formal document update.

Some documents, such as the QAM, require periodic reviews. The QAM is reviewed annually and updated as necessary.

Completed logbooks are numbered, scanned, and the resulting .pdf file stored on the Library drive.

#### **4.1.1.2 External Documentation**

Examples of external documentation include regulations, analytical methods, QAPPs, and client required standards. These documents are maintained by the quality assurance department. When possible, the documents are stored electronically on the Library drive. Instrument manuals are controlled by the individual laboratory areas.

#### **4.1.2 Traceability of Measurements/Documentation Requirements**

A properly designed and implemented documentation protocol will assure that all information presented in an analytical report can be traced back to its point of origin. The documentation protocol must also provide for traceability of non-reported information used to provide supporting value to the analytical report. These items include but are not limited to: stock standard records, test calibration records, data reduction and validation activities, sample custody, facilities monitoring, and final data reporting.

A more detailed review of the documentation procedure and traceability of information is presented in the following sections.



### **4.1.3 Paperwork/Information Flow**

As displayed in Figure 4-2, document flow remains constant regardless of the quality control requirements of the sample. The general axiom is, a COC procedure will fail without a pre-existing scheme of rigid documentation control available. The records trace can provide for the following:

- Answers to questions of analytical integrity
- Assistance in finding and solving random and systematic problems
- Assistance in preventing long term degradation of the analytical process
- Assistance in ensuring continuity of analytical effort despite personnel and mechanical changes

The following subsections identify and describe the procedures followed, and the corresponding documents generated, from project initiation through completion.

#### **4.1.3.1 Project Initiation**

All samples or sample groups entering the analytical process must be accompanied by the appropriate documentation. This documentation is necessary to define the analytical goals and project objectives. Information concerning analytes, reporting limits, and reporting formats must be provided. An inventory of required sample containers must be prepared for each sampling event. This inventory is documented using the Container Packing List (Appendix N).

All projects are initiated through the LIMS system. All documents created during the project initiation phase are maintained and archived to the client filing system.

#### **4.1.3.2 Sample Receipt/Examination**



The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record Logbook (Appendix O). This logbook documents the delivery method, date and time, number of coolers received, client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

Observations on the receipt of each sample delivery group, including sample temperatures, are documented on the “Sample Receiving/Log-in Checklist” (Appendix P). This form was designed in a step-by-step format to walk the log-in technician through all the steps required when receiving and logging-in samples. A supplemental “Sample Receiving/Log-in Checklist Additional Cooler Information” form is available when receiving projects consisting of more than four coolers (Appendix Q).

Additional forms to document sample preservation, “Sample Preservation Verification Form” (Appendix R), and non-conformances, “Sample Receiving Non-Conformance Report” (Appendix S), are also completed.

#### **4.1.3.3 Sample Log-In**

During log-in, a series of computer entry functions are performed in an effort to document and validate the log-in process. The remainder of the Checklist is also used to record the completion of the various steps that must be followed when logging samples into the LIMS system. Once complete, bottle tags are produced and a Work Order generated (Figure 4.3 and Appendix T). The log-in technician will initiate a project or submittal file for each sample delivery group received. This file is labeled with the LIMS system generated project-submittal sequence, and will contain all documents associated with the sample receiving/sample log-in process. These documents will include: all external chain-of-custody forms, sample preservation records, shipping records, any



client correspondence, and a copy of the actual log for each submittal. Upon completion of the analytical process, the project file becomes part of the permanent record of each project.

#### **4.1.3.4 Worklists/Benchsheets**

The worklists and benchsheets produced by the LIMS system are designed to provide the analyst with essential project information. This information not only includes client/project specifications, but also provides an avenue for communication of test specifications and parameter expiration dates and times. This up-front information enables the analyst to make vital decisions in their analytical scheme, and helps to minimize problems after samples are analyzed.

Examples of a laboratory worklists and benchsheets are presented in Appendix U.

#### **4.1.3.5 Management Reports**

Several reports are provided within the TriMatrix Laboratory system to help monitor operational conditions of the laboratory. These reports include: workload reports, on-time reports, and aging logs.

The flow of information from these various reports is geared to a variety of personnel within the management structure of the laboratory, and to specific persons outside the laboratory. Information is generally provided to employees' external of the laboratory for corporate management decisions or in providing information to a particular client about their project.

Examples of management reports are presented in Appendix V.

#### **4.1.3.6 Quality Assurance Reports**



Quality assurance reports play a vital role in the management of the quality system. Quality systems must be closely scrutinized in order to monitor, maintain, adjust, and add, procedures or systems to meet existing and new QA objectives of the laboratory.

Several quality assurance reports are created in this effort. These reports serve different functions and are designed to inform the ultimate user. In the case of a client/invoice report, the quality assurance data is presented to facilitate the objectives of the project requirements from data assessment through full 3rd party data validation.

Quality control reports are also used within TriMatrix to monitor the analytical process and to provide a means by which the analytical process can be viewed over time. Examples of efforts available for this monitoring process are presented in Appendix W.

Quality control reports are used extensively by the laboratory to access the analytical process. Many of these reports are utilized daily to monitor, for example – method accuracy, precision, completeness, and to provide the means for overall data assessment at the batch level. All QC reports are created through the LIMS system.

#### **4.1.3.7 Project Files**

The project file is the comprehensive record of every analytical project completed at TriMatrix. Project files are stored in secure filing cabinets. Items typically retained in a project file include:

- Initial project report/analysis plan/proposal
- All correspondence or documents mailed or received with the samples
- Written record of client phone conversations



- All sample receiving/log-in forms
- Chain-of-custody forms
- Laboratory worksheets
- Copy of the invoice

To save paper and file space, electronic, rather than paper, copies of final reports are typically retained, and can be regenerated on demand.

By default, project files are stored on-site for 1 year, followed by off-site storage at a secured limited access facility for an additional 6 years. Length of storage requirements are determined on a client/project specific basis. If the ownership of the laboratory changes, record storage will become the responsibility of the new owner. In the event the laboratory was to go out of business, each client will be contacted for instructions on record disposition. Client records will be transferred or destroyed as instructed.

#### **4.1.3.8 Quality Control Documents**

##### **A) Instrument Logbooks**

Two different instrument logbooks are maintained, an instrument run-log and an instrument maintenance log. Each log plays an important role in the documentation of daily instrument activities.

The Instrument Run Logbook is used to document all analytical determinations of a designated instrument. These determinations include not only sample analyses, but also recordings of all calibration and calibration runs, quality control analyses, and where applicable, any instrument tuning activities.

The Instrument Run Logbook also provides a chronology of each day's analyses. This chronology plays an important role in the data validation process. All run logs are identified by instrument



manufacturer name, model number, serial number, and the starting and ending dates encompassed. All completed run logs are issued document control numbers, inventoried, and properly archived.

The Instrument Maintenance Log is used to document instrument maintenance procedures, repairs, or modifications. All activities are documented by recording what was done, by whom, and why.

All completed maintenance logs are identified by instrument manufacturer name and model number, instrument serial number, and the dates encompassed. All maintenance logs are issued document control numbers, inventoried, and properly archived.

#### **B) Controlled Temperature Units (CTU)**

Each oven and incubator used for sample processing, and all cold sample and standard storage devices have their temperatures monitored and recorded on a daily basis. Within each CTU is a certified thermometer. Additionally, each CTU used for sample storage, and incubators used for BOD and bacteriological incubation, have their weekend temperature monitored via electronic data loggers. The calibration of liquid and digital thermometers is verified annually.

All temperature readings and thermometer calibrations are recorded in a CTU Logbook. This logbook contains a page for each unit with detailed information on unit identification, serial number, laboratory location, and designated operating temperature. All CTU logbooks are issued document control numbers, inventoried, and properly archived. An example of a Controlled Temperature Log is presented in Appendix X.

#### **C) Balance Monitoring**



Each analytical and top loading balance used at TriMatrix is monitored for accuracy. All daily checks are recorded in a TriMatrix Balance Log (Appendix Y). All balance logbooks are issued document control numbers, inventoried, and properly archived.

#### **D) Standard and Reagent Preparation Logbooks**

All standards and calibration solutions used at TriMatrix are prepared, when possible, from reagents or solutions traceable to national standards. Whether a stock, an intermediate, or a working concentration, each reagent and standard solution is traceable to its origin. This is accomplished using the laboratory's LIMS system and/or a Standard Preparation Logbook (Appendix Z).

Information available on each standard includes:

- The analyte or analytes contained in the standard
- The concentration
- The solvent used to prepare the standard
- The preservative (i.e., nitric acid)
- The date of preparation
- Initials of the preparer
- The expiration date
- The unique identification number

Unique identification numbers are generated by the LIMS system and/or a book, page, and line number system. All standard and reagent preparation logbooks are issued document control numbers, inventoried, and properly archived.

#### **E) Pipet Logs**

All autopipetors utilized for the delivery of standard solutions, diluents, and reagents, are periodically checked for delivery



accuracy. Because these pipetors contain mechanical parts they are subject to inaccuracies if not properly maintained and calibrated.

Daily calibrations (for pipets used to prepare standards), and weekly calibrations (for pipets used to prepare quality control samples) are recorded in a Pipet Calibration Logbook (Appendix AA). Each log is identified by manufacturer name and model number, the pipetor serial number (if available), and the starting and ending dates encompassed. All complete pipet logbooks are assigned document control numbers, inventoried, and properly archived.

#### **4.1.3.9 Confidentiality and Proprietary Rights**

Since significant amounts of information regarding the details of a client's operations are received in the laboratory, it is essential that strict confidentiality be maintained in the handling of all client information. Client data is protected in locked filing cabinets and in limited access computer files. Under no circumstances is the name of a client, or any information regarding that client, revealed to another client or to a regulatory agency without the client's written permission, under penalty of employment termination.

Any details of a client's operations that have necessarily been revealed to the laboratory for testing purposes are considered as proprietary and protected by patents, copyrights, infringement laws, or other legal constraints against disclosure.

#### **4.1.3.10 Document Storage and Traceability**

Archiving of information at TriMatrix has been designed to meet both short-term and long-term storage needs. Archives are maintained for a wide variety of data and documentation. These archives can be categorized into two main groups, a) document



archives (physical documents) and b) electronic archives (data files). Table 1 illustrates the current TriMatrix archival systems, their location, and duration.

Documentation records or logs are maintained for all archival systems to aid in the quick retrieval of information. Extended archival periods or special procedures are also in place for some projects and clients.

#### **4.1.4 Standard Operating Procedures (SOPs)**

Many of the methods published today by various agencies provide only general guidance in performing an analytical determination. A significant part of the variability observed in analytical data is in large part due to minor variations in the analytical process. A Standard Operating Procedure is a guide that clearly defines the exact steps to be followed while performing a procedure. The delineation of these exact steps in an SOP will improve the analytical conditions, which in turn will help the overall reproduction of analytical data.

##### **4.1.4.1 SOP Categories**

SOPs are written for nearly all laboratory activities. The categories utilized in the organization of SOPs are presented in Table 2.

##### **4.1.4.2 SOP Development, Formatting, and Review**

All standard operating procedures are developed and written to the specifications outlined in the TriMatrix guidelines for the preparation of a SOP. These guidelines are presented in SOP format and have been designed to accommodate analytical tests, non-tests such as extractions or digestions, and documentation or non-analytical activities. The guidelines were developed from both USEPA and ASTM protocols for the creation of standard operating procedures.



All SOPs developed by TriMatrix are subject to a review process where signatures or approvals are required from the appropriate area manager, the quality assurance department, and the Vice President of Laboratory Operations. In addition to this overall approval process, each page of an SOP is individually approved by both the laboratory area and quality assurance department (Appendix AB).

SOPs are reviewed and updated as necessary. Minor modifications can be hand edited on the SOP. These modifications must be made through the Quality Assurance Department. Depending on the modification, distribution of the edited SOP (as described below) may or may not be required. All minor modifications will be incorporated into the next revision of the SOP. Major modifications may require the SOP to undergo an immediate formal update.

#### **4.1.4.3 SOP Documentation and Control**

All SOPs are assigned a unique procedure identifier. Other information included in every SOP is the effective date, revision number, information on the author, total number of pages, and identification of any individual page revisions.

All original, approved paper copies of TriMatrix SOPs are controlled by the Quality Assurance department. Approved SOPs are scanned and stored on the network Library drive. This drive is accessible to all laboratory personnel. Copies of all outdated SOPs are destroyed (or marked as obsolete), and the scanned copy of the SOP is removed from the Library drive.

#### **4.1.5 LIMS**



TriMatrix utilizes the Element LIMS system developed by Promium Corporation. This system controls all aspects of laboratory operations. The main functions of the LIMS system are:

- Project Management
- Sample Management
- Work Scheduling and Management
- Data Entry, Verification, and Approval
- Report Generation
- Invoicing

## **4.2 SAMPLE CONTROL, FLOW, AND STORAGE**

Presented in the following section is a description of the policies and procedures that were developed to identify, monitor, and document the flow of samples through the Laboratory. A flow chart depicting this process is presented in Figure 4-3.

### **4.2.1 Project Initiation**

When samples are received at TriMatrix, the necessary information that will direct the analytical scheme has already been developed and implemented within the project initiation/project management process. This process starts with the award of a contract or proposal, a client request, or a pre-scheduled sampling event. The basic steps and supporting documentation involved in the project initiation process begins with the gathering of project information, communications with all affected laboratory areas, and the input of required project related data into the LIMS system. All requests for analytical work are reviewed by the project chemist, and when necessary, applicable management staff to verify the laboratory has the capability to perform the requested tests and meet the requested turnaround times. Requests for changes to in-progress projects must be made with the appropriate project chemist. Changes in methodology will typically require client approval. The project chemist will be responsible for coordinating all requests for changes with the impacted laboratory areas. All approved changes will be formally made via the laboratory's LIMS system, thus continuing the normal paperwork flow.



TriMatrix uses test methods that meet the needs of the client and are appropriate for the tests undertaken. Methods published in international, regional, or national standards are used. TriMatrix uses the latest valid edition of a method unless it is not appropriate or possible to do so. All analytical procedures are documented in SOPs supplemented with additional details to ensure consistent application.

When not specified by the client, TriMatrix will select appropriate methods published either in international, regional, or national standards, by reputable technical organizations, in relevant scientific journals, or as specified by the equipment manufacturer. Laboratory developed methods (or methods adopted by the laboratory) are also used when appropriate for the intended use, and have been validated following the various initial demonstration of capability procedures. When specified by the client, TriMatrix will inform the client if the specified method is considered inappropriate, or out of date.

Routine projects include samples matrixes and analyses that are continuously processed by TriMatrix. Non-routine projects are those that require special analyses, include parameters not routinely run by the laboratory, possess unique holding times, or require expedited turnaround. Non-routine projects will require approval from all affected laboratory areas. This approval process is communicated in several different ways, including everything from the signing of a quality assurance project plan (QAPP) to the transmission and receipt of an electronic mail message.

Occasionally, a portion of a project may involve an analytical methodology not currently possible at TriMatrix. When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. It is preferred that the client specify the subcontract laboratory. When the subcontract lab is not specified by the client TriMatrix will only subcontract to laboratories that are NELAP accredited, or ISO-17025 certified, for the specific method of interest. Client specific program requirements will take precedence over this rule. A registry of subcontract



laboratories used by TriMatrix will be maintained, documenting their NELAP accreditation or ISO-17025 certification.

The development of a project within the laboratory also involves the preparation and shipment of sample collection materials and containers. The processes involved in the procurement, preparation, and shipment of sample collection materials and containers are presented in the sections below.

#### **4.2.1.1 Sample Containers and Materials Procurement**

TriMatrix utilizes only virgin bottle ware for all sample collection kits. All containers are purchased pre-cleaned and come with Certificate's of Analyses.

Specific projects or programs may require the laboratory to verify the cleanliness of the containers. When this is required specific lots will be sequestered from the container vendor. Each lot will be tested to verify the containers meet the project or program requirements. Only containers whose cleanliness has been verified will be used for the project.

#### **4.2.1.2 Preparation of Containers**

All sample containers utilized for the collection and preservation of environmental samples are prepared by the bottle prep group. The staff members of this group focus their activities exclusively in the area of sample container procurement, preparation, and shipping. Project sample container kits are requested using the Container Packing List, presented in Appendix N.

#### **4.2.1.3 Sample Container Shipment**

When all containers have been assembled as requested on the Master Bottle Packing List, the bottles are packaged and placed into one or more shipping coolers. 40 mL glass vials are packed in



small bubble pack bags. An attempt is made to organize each sample cooler to help minimize time spent in the field. When possible this is accomplished by packing bottles together by sample point. When complete, each shipping container will be inspected by a project chemist to verify its accuracy. Documentation of this inspection is made on the bottle packing list. A copy of the bottle packing list is placed in each cooler.

Also provided in each cooler is a set of instructions or comments about the containers, material safety data sheets for all chemical preservatives present, a return address label, an external COC form, and if required, TriMatrix sample bottle custody seals. All materials are packaged in a waterproof zip-lock bag. Examples of these additional materials are presented in Appendix AC.

Packing is now added to the cooler and the shipping container is sealed. When requested, signed TriMatrix custody seals can also be applied to the outgoing cooler.

#### **4.2.1.4 Sample Receipt**

The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record logbook (Appendix O). This logbook documents the delivery method, date, and time, the number of coolers received, the client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

As soon as possible after the shipping cooler is received and all available information entered into the Sample Receipt Record, cooler inspection and sample temperature determination occurs. The observations associated with this step by step process are recorded on the “Sample Receiving/Log-in Checklist” (Appendix P). This Checklist must be completed for all samples for a given project received on a given day. A supplemental “Sample



Receiving/Log-in Checklist Additional Cooler Information” form is available when receiving projects consisting of more than four coolers (Appendix Q).

**IMPORTANT:** When initiating each Checklist, make sure the Receipt Log Page/Line number from the Sample Receipt Record logbook is recorded at the top of each Checklist. This ties the receipt of the sample coolers in with the samples themselves.

Record the cooler number of the first cooler and the current time. Observe and record the type of coolant used. When possible, the sample temperature of three random samples (locations representative of the coolant present in the cooler) will be taken. If a temperature blank was received, measure and record this temperature as well.

Sample temperatures are recorded using a calibrated infrared thermometer. Because this type of thermometer is actually measuring the temperature of the container, it is critical that the temperature is taken as the sample is removed from the cooler. The container warms up quickly and any other method will result in an incorrect reading. Do not dry the container prior to measuring the temperature. Containers wet from melt water are preferred to dry containers. Record the temperature values on the Checklist. Report all temperatures to the nearest 0.1° C. If a correction factor is necessary, record the correction factor and the corrected temperature on the Checklist. If any temperature exceeds 4° C, average the three sample results and also report the average. If the average temperature of the three samples, or the temperature of the temperature blank exceeds the 6° C required by most regulatory bodies, it must be noted on the Checklist.

If sample receipt and temperature determination occurs outside of normal business hours, place received coolers in the walk-in for



storage. Assemble all the paperwork, and place it in the after-hours basket. The remainder of the receiving process will be performed by a log-in technician during the next business day.

#### **4.2.1.5 Sample Examination**

Samples received at TriMatrix are required to be accompanied by a TriMatrix Laboratory Chain-of-Custody (COC) form (Appendix AD). For samples received without this form, the log-in technician will initiate the COC process. Should a submittal or delivery group be identified as an internal COC project, the log-in technician will initiate the procedures outlined in section 4.2.2 B.

The remainder of page 1 of the Checklist is now filled in. Observations are made on the accuracy of the COC and the condition of the sample containers. Many of the aqueous samples received have been subjected to some form of chemical preservation. Verification of the preservation is required; however, depending on the analysis this verification may not occur during the log-in process. The “Sample Preservation Verification Form” (Appendix R) specifies what container types will have their preservation verified during log-in. The form also specifies what container types can have an incorrect preservation adjusted. Preservation verification is performed via a pH check using calibrated pH strips. Determine the correct reading against the color chart on the pH strip container. Document the pH found on the Sample Preservation Verification Form. Use only the pH strips located in the log-in area whose calibration has been verified and recorded in the pH Strip Calibration Logbook (Appendix AE).

Should a) the result of any preservation check indicate that the sample has not been properly preserved in the field (or the buffering capacity of the sample has resulted in an unacceptable sample pH at receipt) or b) there is insufficient evidence indicating that other needed preservation reagents (e.g., Zinc Acetate for



Sulfides) have been added, then a Sample Receiving Non-Conformance Report (Appendix S) is to be initiated and the project chemist contacted as soon as possible. In some instances, the holding time of such samples may be shortened. No preservation adjustment may be made without approval from a project chemist.

**IMPORTANT:** Shaded boxes on the Checklist indicate an out-of-control situation. The selection of any shaded box during the completion of this form also requires the initiation of the Sample Receiving Non-Conformance Report.

Collect all paperwork and deliver to the appropriate project chemist for review. Any issues that require contact with the client for resolution will be made in a timely manner. The project chemist will create a submittal and return the paperwork. Once the project chemist returns the paperwork, page 2 of the Checklist can be completed, and the samples logged into the LIMS system.

#### **4.2.1.6 Sample Log-In**

All samples received by TriMatrix are logged into the LIMS system. The log-in procedure assigns a unique TriMatrix sample number to each sample, allowing samples to be tracked, data stored, and quality control associated for any sequence of events during a particular analytical period. The primary steps involved in the sample log-in process are presented below.

#### **4.2.1.7 Sample Splitting**

In the event that TriMatrix is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the log-in technician can provide sample splitting services; however, sample splitting will typically be performed by a laboratory area chemist. These services include taking the sample as received and



sub-sampling it into the appropriate bottle with the preservative requirements as set forward in Appendix AF – Sample Collection Guidelines Bottle and Preservative Requirements. Sample splitting will only be performed when instructed by a laboratory project chemist with client approval.

#### **A. Sample Splitting-Water Samples**

Laboratory area managers will be consulted in order to insure that sufficient volume will be available to all areas of the lab after splitting. In the event that sufficient volume does not exist, the Project Chemist will be immediately notified for resolution.

When a bulk sample arrives for both organic and inorganic analysis, and sufficient sample exists, the organic aliquots will be removed first. The remainder of the sample will be transferred to properly preserved containers for each inorganic analyses.

#### **B. Sample Splitting-Solid Samples**

When solid samples, such as sediment or soil, are to be received at TriMatrix, every attempt will be made by the Project Chemist and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic area and the other to the inorganic area. If only one sample is received and if organic analyses are required, the organic aliquots will be removed first. Prior to sub-sampling, solid samples will be made homogeneous by either one or all of the following manners:

- Stirring
- Grinding
- Particle separation (sieving)



The laboratory area manager is responsible for deciding how a solid sample will be split. Problems or concerns that may arise on splitting a solid sample will be addressed by the Project Chemist and Laboratory Area Manager. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting.

#### **4.2.1.8 Sample Distribution**

All samples received at TriMatrix are labeled by the log-in technician. These labels include both the necessary information for proper identification, and information on any potential for flammability, reactivity, contact, or health based risks.

After completing the log-in process of all the various samples connected with a particular project, the log-in technician will store the samples in the correct Controlled Temperature Unit (CTU).

- Routine Water and Solid Samples: Samples that require refrigeration will be stored in the CTU designated for all routine water and soil samples.
- Routine Volatile Water and Solid Samples: All volatile samples are stored in designated VOA CTUs. Volatile water and soil samples are segregated and stored separately. No other sample types are stored in the VOA CTUs.

All CTUs used for VOA sample storage will also contain a storage blank. The storage blank is a preserved 40 mL VOA vial filled with deionized/distilled water. The storage blank is replaced and analyzed on a weekly basis. If positive results are observed for any target analyte above the laboratory's minimum reporting limit, all samples stored concurrently in the CTU must be evaluated for possible contamination. All sample results



within 5 times the level quantitated in the storage blank must be qualified as estimated.

- **Odoriferous and Hazardous Samples:** Stored separately in a special vented facility. If volatile analyses are to be performed, samples are stored under refrigeration. Samples are identified to the laboratory by means of a narrative within the LIMS System.

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under COC procedural safeguards.

#### **4.2.2 Chain-of-Custody (COC)**

All samples received by the laboratory require some form of chain-of-custody (COC). TriMatrix practices two levels of COC, external and internal. The degree of custody tracking and documentation is driven by the final disposition of the laboratory data. Generally, if samples and their analytical results are subject to involvement as physical evidence or in a legal procedure, both external and internal custody procedures will be followed. If samples or results are not subject to legal procedures, only external COC procedures will be followed. A description of these two custody scenarios is presented as follows:

##### **A. External COC**

Samples only requiring external COC will have their custody tracked from sample collection to delivery at the laboratory. This process involves the completion of a TriMatrix external COC form, as presented in Appendix AD. This form accompanies the sample containers prepared by TriMatrix to the sample collection site. Any sample or submittal received at the laboratory without a TriMatrix external COC form will initiate a process where the log-in technician will complete the necessary external COC forms for carrier sign-off.



For document control purposes, all external COC forms have a unique identification number.

#### **B. Internal COC**

Samples requiring strict COC will initiate the process by which all events or periods of sample handling will require a traceable document protocol.

The internal COC process involves the completion of a TriMatrix internal COC form for all phases of the analytical process. This includes sample extractions, distillations, digestions, analyses, and disposal. An example of the TriMatrix internal COC form is presented in Appendix AG. All internal COC forms are maintained in a series of submittal or delivery group folders.

#### **C. Sample Security**

All samples, whether under external or internal COC protocols, are maintained in a limited access secured area. This level of security is applied to all phases of the analytical process from sample log-in to final sample disposal.

#### **D. Sample Disposal**

All samples received are subject to disposal as waste once tested and discarded. Three general categories discarded samples fall into are the following:

1. A sample may be returned to the client (specifically, if highly contaminated).
2. Too contaminated for municipal disposal and must be disposed of as waste through a hazardous waste facility.
3. Inert, uncontaminated, and nontoxic samples in accordance with municipal waste regulations may be disposed of in the municipal dumpster and/or the laboratory waste room sink leading to the city sewer.



#### **4.2.3 General Laboratory Security**

Access to the laboratory is handled in a secure fashion, with access restricted to authorized personnel only. All laboratory areas including sample storage, sample container preparation, analytical laboratories, sample preparation, sample disposal, analytical documents, and data files are restricted. Non-authorized personnel may enter these areas only when escorted by a laboratory staff member.

It is the responsibility of all laboratory staff members to insure that the rules of restricted access are followed and maintained at all times.

### **4.3 CALIBRATION AND CALIBRATION VERIFICATION**

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment used in conducting laboratory analyses. Calibration of the instruments and equipment is performed prior to each use or on a scheduled periodic basis.

Calibration of laboratory instruments and equipment is performed to verify that the analysis portion of the testing process is functioning properly and at the required sensitivity. A calibration section included in each analytical SOP covers the frequency, stability, and specific calibration steps, based on analytical method requirements and instrument or equipment manufacturer's recommendations.

Initial calibration is performed using standards of certified value to establish the linear range of the analysis for the analytes of interest. Each calibration curve is verified using a Second Source Calibration Verification Standard (SCV) prepared from a source dissimilar to that used in the preparation of the calibration standards. The calibration is also verified at the beginning and during the analytical sequence, using a standard prepared from the same source as that used in the initial calibration.

Calibration activities are divided into three categories:

Field Equipment (section 4.3.1)



Laboratory Instrumentation (section 4.3.2)

Laboratory Equipment (section 4.3.3)

#### **4.3.1 Field Equipment**

Perform daily calibration checks on field equipment prior to the commencement of any field analyses. Follow the written calibration procedure for each individual piece of field equipment. The equipment is held out of service until repairs and successful recalibration occurs. A summary table of all calibration procedures and frequencies is included (Table 3).

#### **4.3.2 Laboratory Instrumentation**

Calibration of laboratory instruments is based on approved SOPs. Records of calibration, repairs, or replacement are filed and maintained by the designated laboratory analyst. These records are filed at the location where the work is performed and are subject to QA audit. For all instruments, the laboratory maintains in-house spare parts or service contracts with vendors. A summary table of all calibration procedures and frequencies is included (Table 4). Flag any instrument that does not pass daily requirements. Hold the instrument out of service until repair or successful recalibration occurs.

##### **4.3.2.1 Inorganic/Classical Chemistries**

Inorganics analysis utilizes a wide variety of wet-chemical procedures and instruments. Calibration steps may vary depending on the specific analytical method being utilized. However, certain general principles of calibration apply to all inorganics testing. Every analytical method requires calibration or calibration verification prior to sample analysis. Using a group of certified standards, the linear range is defined. The calibration is checked on a continuing basis to be certain that the method is within the required test parameters. All inorganic calibrations must meet the specific requirements described below unless required otherwise by the method or manufacturer.



The instrumentation used to conduct these analyses is calibrated using calibration standards prepared by dilution of stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve.

An SCV originating from a dissimilar stock solution than that used for preparation of the calibration standards is prepared and analyzed. Continuing Calibration Verification blanks and standards (same source as that used in the initial calibration curve) are run at the beginning, and periodically, throughout the analytical sequence, typically after every 10 analyses. The value of the continuing calibration standard concentration must agree within the method specified criteria; generally  $\pm 15$  percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous 10 samples.

#### **4.3.2.2 AAS/ICP/MS Emission Systems**

The atomic absorption spectrophotometer (AAS), inductively coupled plasma emission spectrophotometer (ICP), and inductively coupled plasma mass spectrometer (ICP/MS) instruments are calibrated by the use of a minimum of three calibration standards (6 for ICP/MS) prepared by dilution of certified stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration



standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Calibration standards contain acids at the same concentration as the digestates. A continuing calibration standard is analyzed after every 10 samples. The value of the continuing calibration standard concentration must agree within method specified criteria, generally  $\pm 10$  percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous ten samples.

#### **4.3.2.3 Gas/Liquid Chromatography**

Analysis performed by gas chromatography follows USEPA protocols. The instrument is calibrated using three or five point calibration curves (depending on method requirements) for both volatile and semi-volatile compounds. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Continuing calibrations are performed after every ten samples. The value of the continuing calibration standard must agree within  $\pm 15$  or 20 percent (depending on method requirements) of the initial value or the appropriate corrective action is taken, which may include recalibrating the instrument and must include reanalyzing the previous ten samples.

#### **4.3.2.4 Gas Chromatography/Mass Spectrometry (GC/MS)**

Prior to calibration, the instruments used for GC/MS analyses are tuned by analysis of p-bromofluorobenzene (BFB) for volatile analyses and decafluorotriphenylphosphine (DFTPP) for semi-



volatile analyses. Once the tuning criteria for these reference compounds are met, the instrument is initially calibrated using a three or five point calibration curve (depending on method requirements). The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must still be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. The instrument tune will be verified each 12 or 24 hours of operation (depending on method requirements). Continuing calibration is verified as specified in the method. The calibration standards are commercially available certified standards containing the target analytes, surrogate spikes, and internal standards.

#### **4.3.3 Laboratory Equipment**

Personnel performing calibration should also be alert for any condition that renders a piece of equipment inoperable or unfit for use; for example, inspect thermometers to ensure that mercury or alcohol columns are not separated. If an equipment malfunction is noted during calibration, the equipment must be tagged and removed from service. The equipment is held out of service until repairs and successful recalibration occurs. Record all malfunctions, repairs, and re-calibrations in the appropriate logbook.

Maintain records for each piece of equipment requiring calibration, showing equipment description and identification number, calibration frequency and acceptable tolerances, personnel performance calibration, date, reference material used, calibration results including acceptance or failure, removal from service, repairs, and date and authorization for return to service.

##### **4.3.3.1 Balances**

An annual third party maintenance and calibration is performed on all balances. Daily calibration is performed by TriMatrix on all



balances using class S or higher NIST traceable weights. Provided daily calibration is successful the weights themselves are indirectly calibrated on a daily basis via the third party's calibration; therefore, re-certification or replacement of the weights is not required every five years.

#### **4.3.3.2 Thermometers**

Thermometer calibration is performed annually, using a NIST certified thermometer. The NIST thermometer must be re-certified or purchased new every five years. Written records are maintained of all annual calibrations.

### **4.4 DATA REDUCTION, VALIDATION, AND REPORTING**

Data reduction is the process by which raw analytical data is tabulated and calculated. Data validation is the review of the data generation and reduction process. Data reporting is the compilation of all sample results for distribution to the client. All analytical data generated by TriMatrix Laboratories is subjected to the reduction, validation, and reporting process as described below.

#### **4.4.1 Laboratory Data**

##### **4.4.1.1 Data Reduction**

Initial results for most analyses are calculated using a computer directly interfaced to the instrument. Data reduction is accomplished using software that has been validated for its intended purpose. The initial result is exported to the LIMS system. Data such as initial volume, final volume, and percent solids, are used by the LIMS system to calculate a final result. When manual data reduction is required, it is performed according to the written standard operating procedure for that analysis.

##### **4.4.1.2 Manual Integrations**



Manual integration is defined as any post acquisition adjustment to the automated software peak integration. Manual integrations are often times legitimately required to correct for baseline drift, noisy baselines, poorly resolved peaks, closely eluting or missed peaks, peak tailing, or peak splitting. Manual integration may never be used for the sole purpose of correcting failing quality control parameters (i.e. shaving or enhancing peak areas or heights to make failed calibrations, surrogates, or internal standards pass), or as a substitute for poor or ineffective sample cleanup. Manual integration must be used cautiously due to the increased scrutiny inherent with adjusted data. Particular attention will be paid to manual integrations performed on standards and blanks since these samples are typically free of interferences.

Before and after documentation must be provided with all manual integrations. This documentation must clearly show the original integration “before”, and the manual integration “after” baseline. Clear identification of manual integrations must be included in the case narrative for all samples analyzed under Federal Facilities work requirements. All quantitation reports must clearly identify manual integrations by flagging the peak with a designator that cannot be removed by the analyst. Additional documentation requirements include:

- Date of the manual integration
- Reason for the manual integration
- The integration area or height before manual integration
- The integration area or height after manual integration
- A signature/date by both the analyst and the reviewer.

Any questions concerning manual integration must be resolved with the area manager or the quality assurance officer before final results are approved and released to the Project Chemist. The



complete laboratory manual integration requirements are detailed in the TriMatrix manual integration SOP GR-10-115.

#### **4.4.1.3 Four Levels of Data Validation**

##### First Level Review

Data validation begins with the analyst. It is the basic responsibility of the analyst to produce data that is complete, correct, and conforms to all applicable methods and standard operating procedures. If results are not acceptable, it is the duty of the analyst to perform the appropriate corrective action and to thoroughly document that action. The analyst will verify the following before updating the analysis status to “Analyzed”:

- Applicable standard operating procedures were followed
- Proper analytical sequence was followed
- Sample preparation information was correct
- Calibration has been performed properly
- Analytical results are complete
- Holding times have been met
- Method criteria were met
- Any special sample preparation or analytical requirements have been achieved
- All analytical abnormalities have been noted
- Corrective actions are thoroughly described
- Good record keeping practices have been followed
- Any problems are communicated to area manager
- Data was correctly transferred to Element
- Calculations were performed properly
- Quality control samples are within established limits
- Documentation is complete
- Raw data, including chromatograms and instrument printouts are complete
- Case narrative or qualifier pages are complete



### Second Level Review

A laboratory area peer or designated validator, in essence, performs the same validation steps performed by the analyst. Particular attention should be paid to:

- Dilution factors were entered correctly and detection limits elevated accordingly
- Analysis dates are correct
- Quality control and analytical batch information is correct
- Quality control results and spike amounts are correct and in control
- Project specific limits are correct
- Run a draft copy of the report, specific to the laboratory area, to verify all results have been adjusted correctly
- Any required qualifiers or narratives have been entered

Any problems must be resolved with the analyst, and when appropriate the quality assurance manager, prior to updating the status to “Reviewed.”

### Third Level Review

Once all analyses associated with a work order have been entered into the LIMS system and approved, the project chemist will perform the Third Level Review. This review will verify that:

- The requirements of the client have been met
- All required narratives and qualifiers have been included
- All quality control parameters required are in the report
- Results of complimentary tests make sense
- The data is accurately presented
- Holding times have been met
- Calibration checks are sufficient



- Documentation is complete

Once this review is complete the project chemist will approve the data and generate a final report. It is during this time that any data package deliverables are collected and reviewed. When printed the work order status updates to “Reported.”

#### Fourth Level Review

The project chemist will perform a final review of the data package hard copy to ensure that:

- All required data package components are complete and accounted for
- Quantitative results are correct
- The overall presentation of data to the client is in an understandable format

In addition to the formal data validation guidelines listed above for the analyst, area manager, and project chemist, there are many practical questions that all of these persons need to keep in mind when reviewing data and finished client reports. Among these “common-sense” evaluations of laboratory data are the following important considerations:

- Data makes good, sound, practical sense
- Multiple runs of the same samples relate, match, or are within acceptable range
- Data from complimentary analyses compares, i.e. COD>BOD>CBOD
- Total cyanide  $\geq$  amenable and free cyanide
- Total solids  $\geq$  suspended and dissolved solids
- TKN  $\geq$  organic N + ammonia N
- Inorganic N = ammonia N + nitrate N + nitrite N
- TOC < BOD or COD



- Total phosphorus  $\geq$  ortho phosphorus
- Calculated total dissolved solids/conductivity = 0.55 – 0.7
- Analytical run looks good; proper decisions were made
- Peaks from chromatogram or instrument printout look normal
- Computer identifications are correct
- Are qualitative/quantitative results real, especially low level
- Know and be sensitive to common laboratory contaminants
- Know area/analytical method pitfalls-be extra cautious
- All practices are sound and are supported by documentation-no appearance of random decisions

When complete the report will be signed. Data packages with deliverables will be scanned and archived. Work order status will be updated to “Completed”.

#### **4.4.2 Field Data**

All data reduction, validation, and reporting for field activities must meet the same requirements as those required in the laboratory. Many of the field instruments, such as those measuring pH, dissolved oxygen, turbidity, temperature, and specific conductance, require a manual data printout from a computer interface. The analyst is responsible for immediate tabulation and calculation of raw data in the field. The field section manager must perform a prompt, on-site validation of field data before the opportunity is lost to perform any necessary field re-tests.

#### **4.4.3 Subcontracted Data**

Analytical results from subcontracted samples will be reported as an attachment to the TriMatrix data package. The attachment will contain the entire subcontracted data package as received by TriMatrix. To eliminate the impression that the subcontracted analyses were performed by TriMatrix, subcontracted results will never be incorporated into the TriMatrix generated report.



## **4.5 VERIFICATION PRACTICES - EXTERNAL/INTERNAL QUALITY CONTROL**

### **4.5.1 Standard Reference Materials**

A crucial step in the generation of quality data is the purity and traceability of reference materials used in the analyses. Reference materials may be physical standards (such as certified thermometers and weights used to calibrate laboratory thermometers and balances) or chemical standards (used to establish and check operational calibration of analytical methods). Physical standards should be traceable to the National Institute of Standards and Technology (NIST). Physical standards must be recalibrated (by an external vendor certified to perform the calibration), or purchased new every five years. Chemical reference materials of high quality can usually be obtained from reliable commercial vendors. For a given analysis, standard reference materials must be kept on hand from more than one vendor source. During the testing operation, standard reference materials from different vendor sources are crosschecked with each other.

### **4.5.2 Internal Quality Control Programs**

TriMatrix routinely adds samples to the sample stream to demonstrate the total testing process is operating within prescribed limits for accuracy and precision. With the exception of Blanks, the concentration of these quality control samples is known prior to the analysis. Types of Quality Control Samples are presented in Table 5. Duplicates and spiked duplicates are selected at random, and when not specified are rotated among clients.

### **4.5.3 External Quality Control Samples-Proficiency Testing**

TriMatrix Laboratories receive Performance Testing (PT) samples on a scheduled basis from state and federal regulatory agencies as well as certain client organizations. A summary of these PE samples is given below:

<b>PT Program</b>	<b>Sample Type</b>	<b>Source</b>	<b>Frequency</b>
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WS	Drinking Water	ERA	Semi-Annual
WP	Waste/Ground Water	APG	Semi-Annual
Soil	Soil	ERA	Semi-Annual
Varies	Environmental	State/Federal Programs	Varies
Varies	Environmental	Client	Varies

TriMatrix receives written reports from sponsoring agencies grading not only their performance, but also a comparison to other laboratories participating in the study. This provides feedback to laboratory personnel regarding the satisfactory use of analytical methods and equipment. Additionally, results from all single and double blind PT samples are used as part of the laboratories fraud prevention and detection program.

## **4.6 DATA ASSESSMENT PROCEDURES**

### **4.6.1 Precision**

Precision of laboratory analyses will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) for organic analyses, and laboratory duplicate or MSDs for inorganic analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses using the following equation:

$$\%RPD = \left( \frac{S - D}{\frac{S + D}{2}} \right) \times 100$$

where:

S = first sample value (original of MS value)

D = second sample value (duplicate or MSD value)

### **4.6.2 Accuracy**



Accuracy of laboratory results will be assessed for compliance with the established QC criteria using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, equipment blank, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using the equation below:

$$\%R = \left( \frac{A - B}{C} \right) \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the unspiked sample

C = the amount of the spike added

#### **4.6.3 Control Limits**

Unless fixed in the analytical method, all quality control acceptance limits in use at TriMatrix are derived from historical data, for each method, matrix, and QC type combination. Precision and accuracy control limits are calculated at a 99% confidence level (+/- three standard deviations); warning limits are calculated at a 95% confidence level, (+/- two standard deviations). Accuracy windows are calculated using the mean of the percent recoveries. Precision windows are calculated as specified in SW-846, using the relative percent difference of the amounts found, not the percent recoveries.

#### **4.6.4 Uncertainty**

In addition to the precision and accuracy of a result, a value relating to confidence is available in the form of a measurement uncertainty estimate. The measurement uncertainty value is estimated using the QC-based nested approach and is calculated at the 95% confidence level. Uncertainty estimates are reported as "percent relative uncertainty."



#### 4.6.5 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated as follows:

$$\text{Completeness} = \left( \frac{\text{valid data obtained}}{\text{total data planned}} \right) \times 100$$

### 4.7 PROCEDURES FOR CORRECTIVE ACTION

When a non-conforming event or process deviation has occurred, corrective action is required. A written standard operating procedure (plan for corrective action) provides the steps for dealing with an out-of-control testing situation. The assessment of whether the process is out-of-control is based on predetermined limits for laboratory operations. Non-conformances based on statistical analysis or quality control samples are readily apparent and easy to identify. A process deviation, which does not have a directly observable impact on data quality, is more difficult to discern. Examples of the latter, subtler types of non-conformances include volatile samples not properly stored; oily layers in certain types of samples that may interfere with analysis; or a water-soaked sample label whose information is barely legible. Discovery of a non-conforming event or process deviation can result from the observations of a staff member, a review of laboratory data at any level, the result of an audit, or a client complaint. A corrective action investigation will be initiated within one week of the discovery of any non-conformance. The time frame required to resolve a specific deficiency and implement the corrective action is dependant on the magnitude of the problem and the defensibility and use of the data. Most non-conformances should be resolved within 60 days from the initiation date. Non-conformances that specifically impact sample results should be resolved within 14 days.

**NOTE:** The client must be contacted within 48 hours (2 business days) upon the discovery of any event that may cast doubt on the validity of a sample result.

The overall scheme of a corrective action plan can be outlined as follows:

1. Define the problem and evaluate the significance of the non-conformance;



2. Assign responsibility for evaluating the problem and determine if the client should be notified and/or work recalled;
3. Determine thorough investigation of all the pertinent facts what the probable cause of the problem is;
4. Select and implement the action(s) most likely to eliminate the problem and prevent recurrence;
5. Assign responsibility for carrying out the corrective steps and implement the action;
6. Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.

Specific responsibility for implementing corrective action is as follows:

It is the responsibility of the analyst or other employee who observes a non-conforming event to:

- Identify and define the problem.
- Fill out a Non-Conformance Investigation Report (refer to Appendix AH).  
When applicable, investigate and attempt to determine the cause of the problem. Report the problem promptly to the area manager. When applicable, accept responsibility for implementing the corrective action approved by the area manager.
- When applicable, evaluate the effectiveness of the corrective action.
- When applicable, verify that the corrective action has eliminated the problem.

It is the responsibility of the laboratory area manager to:

- Review the problem and the proposed corrective action.
- If the reporting person does not have a remedy, work together with the person to determine a satisfactory solution.
- Assign the final corrective action steps to be performed.

It is the responsibility of the QA Department to:

- Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.
- Review, sign, and categorize every Non-Conformance Investigation Report.
- Randomly review corrective action documentation in laboratory through internal audits to ensure that adequate records are being kept.



The ultimate goal of every non-conformance investigation is to resolve the error through identification of the error's root cause. Ideally, once the source of error is found, change can be implemented to prevent reoccurrence of the same error thereby providing a system of continuous quality improvement.

Non-conformances can originate from anyone in the laboratory. Provide the QA department with a copy of the initial report at the time of its distribution, followed by a copy of the completed report. The final report will be distributed to all necessary personnel. Initiation of non-conformance reports associated with out-of-control PT samples will commence with the QA department. The initial non-conformance will be typed up and may include attachments such as a graph charting the history of PT results for that analyte. The history of results for that analyte in PT studies will also be reviewed through the database, looking at additional items such as method, matrix, analyst, vendor, and study type (WP, WS, etc.).

**NOTE:** Non-conformances associated with PT samples must be completed and distributed to state, federal, and other applicable regulatory agencies within the time frame established by that agency.

Returned non-conformance reports will be typed and the final report may include copies of raw data, information concerning traceability, graphs charting historical data, graphs charting trends in analysis, calibration graphs, or any other information relevant to the investigation.

When investigating a failing PT sample, a questionable analytical result, or a client complaint, the following systematic approach for error analysis should be followed until the primary source of error is located and resolved. Progress through them in the order they are presented below (easy to determine transcription error through difficult to determine analytical/procedural failure).

1. Consolidate all necessary raw information, run data and associated calibration and quality control data for both the reported and any non-reported analyses of that sample.
2. Confirm that the intended result was the reported result (transcription error).
3. Verify that the sample was prepped correctly.



4. Verify the correct analytical and pre-treatment method was used.
5. Double check all manual calculations, looking for incorrectly calculated results, missing dilution factor, wrong initial and final volumes, etc. Where possible manually calculate the result and compare with the reported result.
6. Compare the age of the calibration to the PT analysis date.
7. Review data associated with all quality control samples for biases. Also evaluate all QC solutions with respect to age, source, storage, and handling.
8. Determine the reasonableness of the data. Verify that all QC parameters were in control. Compare results to established limits to the data quality objectives of the study (i.e. tighter QC required for WS studies).
9. Review standard laboratory techniques used on the sample and all associated QC analyses. Were measurements used in quantitation made volumetrically? Were pipets and volumetric flasks used, or were less stringent techniques employed? Were serial dilutions made during the preparation of the curve?
10. Review analytical conditions, integration, background corrections, analyte resolution, and any confirmation runs.
11. Review calibration ranges. Are they too large for the analysis? An over extended calibration range will appear S-shaped. Check the population of curve points in the area of the analyte concentration.
12. Review calibration type (linear, average, response factor, polynomial non-linear, etc.). Reprocess multi-level curve data through a best fit program and if linear, perform a residuals analysis to identify outlier calibration points. If the result was quantitated using an average response factor, compare with the best-fit information and confirm justification for use of the average response factor quantitation.

In general, there are three major areas where corrective action is required. These categories are described below. Non-Conformance Reports are required on indications flagged with a \*. Other indications may require a Non-Conformance Report based on the circumstances.

#### **4.7.1 Quality Control Failures**

These are usually handled within the laboratory by the analyst.

#### Indications of Non-Conformance



- Blanks, laboratory control, or spiked samples contain contamination greater than acceptable levels.
- Suspicious trends in spike recoveries or relative percent differences (RPD) between duplicates.
- Initial instrument blank, initial calibration standards, QC check standards, continuing calibration standard spikes, or method blanks are outside acceptance criteria.
- The method blank or instrument blank analysis exceeds the detection limit for the analyte.

#### Recommended Corrective Action

- Prepare another instrument blank. If the response is still greater than the reporting limit, look for sources of contamination in reagents, the laboratory working environment, and the instrument.
- Reanalyze standard. If results are still unacceptable, prepare new standards. If necessary obtain new primary standards.
- Reanalyze continuing calibration standard. If necessary, recalibrate and reanalyze samples since last successful continuing calibration.
- Evaluate preparation of spikes, spiking techniques, spiking equipment and materials.

#### **4.7.2 Procedural Failures**

These are usually handled by the laboratory area manager and the quality assurance department.

#### Indications of Non-Conformance

- There are unusual changes in detection limits.
- Statistical quality control data is demonstrating unacceptable trends or is outside the warning or acceptance limits.
- Deficiencies are evidenced on performance evaluation samples or internal or external audits.
- Clients express concern about the quality of their data.

#### Recommended Corrective Action



- Review the method with the analyst.
- Reanalyze the samples and evaluate the results.
- Recalibrate the instrument or analysis method with freshly prepared standards and reanalyze the samples.
- Re-extract and reanalyze the samples per the method.
- Evaluate the data and sample behavior and investigate any possible chemical interferences.
- Re-run the samples using the method of standard additions.
- Check the instrument for possible maintenance deficiencies.
- Seek additional help from other analysts or provide additional training for personnel involved.
- Perform a system audit to evaluate corrective action measures.

#### **4.7.3 Test Specification Failures**

These are usually handled by the analyst, laboratory area manager, and the quality assurance department.

##### Indications of Non-Conformance

- Quality control check standard data is outside the acceptance limits defined for that analyte.

##### Recommended Corrective Action

- Review the method with the analyst.
- Reanalyze the check standard and evaluate the results.
- Prepare fresh check standard or new primary standard.
- Recalibrate the instrument or analysis method.
- Switch to a different standard vendor.
- Investigate possible chemical interferences.
- Check the instrument for possible maintenance deficiencies.
- Retrain the analyst.

#### **4.7.4 Customer Complaints**



The Quality Assurance Department coordinates with the client services staff to receive quality feedback from clients. It is the responsibility of the QA department to communicate any customer complaints to the laboratory operating areas and to follow-up on corrective action taken to prevent a recurrence.

#### **4.8 PROCEDURES FOR PREVENTIVE ACTION**

Changes and enhancements to existing policies and procedures are not always made based on the result of failing analytical performance or other non-conformances. Borderline performance, equipment changes/modernization, or outdated internal procedures are all areas that may require modification or enhancement. Employees are encouraged to analyze internal procedures of all kinds, and offer suggestions for improvement. A Preventive Action Investigation form exists for this purpose (Appendix AI). The form is used to record a description of the existing procedure and a proposed solution, an action plan and systematic implementation schedule, and a follow-up section to monitor the effectiveness of any resulting changes.

All Preventive Action Investigations are loaded into a database similar to that used to track non-conformances

#### **4.9 DEPARTURE FROM DOCUMENTED PROCEDURES**

##### **4.9.1 Management Policies**

Any departure from a laboratory written standard operating procedure not directly involving sample analysis or processing must be approved by the area manager. The area manager must file a Non-Conformance Investigation Report. The Non-Conformance Investigation Report must be included as part of the data package.

Any departure from a SOP involving sample processing or sample analysis must be justified in writing by the analyst and laboratory area manager. The prior written approval of the laboratory president must be received before performing the analysis. The laboratory president must also file a Non-



Conformance Investigation Report. This Non-Conformance Investigation Report must be included as part of the data package (the exception to this requirement is those items in the analytical methods where a written justification for technical and scientific reasons has been determined by the analyst and approved by the Laboratory President as a deviation from the analytical method).

#### **4.9.2 Method Modification and Variances**

Modification of, and variances in, analytical methods, except for the deviations justified in writing and approved per section 4.9.1, are strictly prohibited.

### **4.10 PERFORMANCE AND SYSTEM AUDITS**

#### **4.10.1 Internal Audits**

Annually the laboratory will be audited by the quality assurance department to verify compliance to ISO-17025 and various State and Federal requirements. Additionally, quarterly internal audits will be conducted by the quality assurance department. Together these audits will encompass all elements of the quality system. A formal written follow-up will be conducted after every internal audit to verify that any deficiencies cited have been corrected, and that the corrective actions have been successful. The following areas will be included in the required internal audits.

##### **4.10.1.1 System Audits**

System audits are used to determine that each component within a laboratory system is functioning properly and adheres to the appropriate standard operating procedures, analytical methods, and requirements of the Quality Assurance Manual. Systems to be audited include:

- A). Sample Handling and Control
- B). Sample Analysis



- C). Records Processing and Control
- D). Support Systems (such as air handling, DI water, analytical balances, raw materials, etc.)

If during the course of an internal audit, problems were uncovered that may have impacted the laboratories ability to generate quality data, written notification must be provided to all impacted clients. Impacted clients include all those clients who received results from samples analyzed during the time frame the problem occurred. This is accomplished by a letter explaining the problem, and includes revised copies of the report that, if necessary, include any required data qualifiers.

#### **4.10.1.2 Documentation Audits**

The Quality Assurance department also performs audits of the laboratory documentation (laboratory notebooks, benchsheets, instrument run logs, client file folders, etc.) to assess the thoroughness and completeness of the documents.

#### **4.10.1.3 Surveillance Audits**

The Quality Assurance department, Area Manager, or their designate observes an analyst in detail as a test is being performed. Attention is given to general laboratory demeanor (orderliness, cleanliness, good laboratory practices in measuring, documentation, etc.) as well as to adherence to analytical methods and standard operating procedures.

#### **4.10.1.4 Quality Assurance Reports to Management**

The Quality Assurance Manager provides the Laboratory President with a copy of all external audit reports. The report details any deficiencies identified as well as recommended corrective actions.



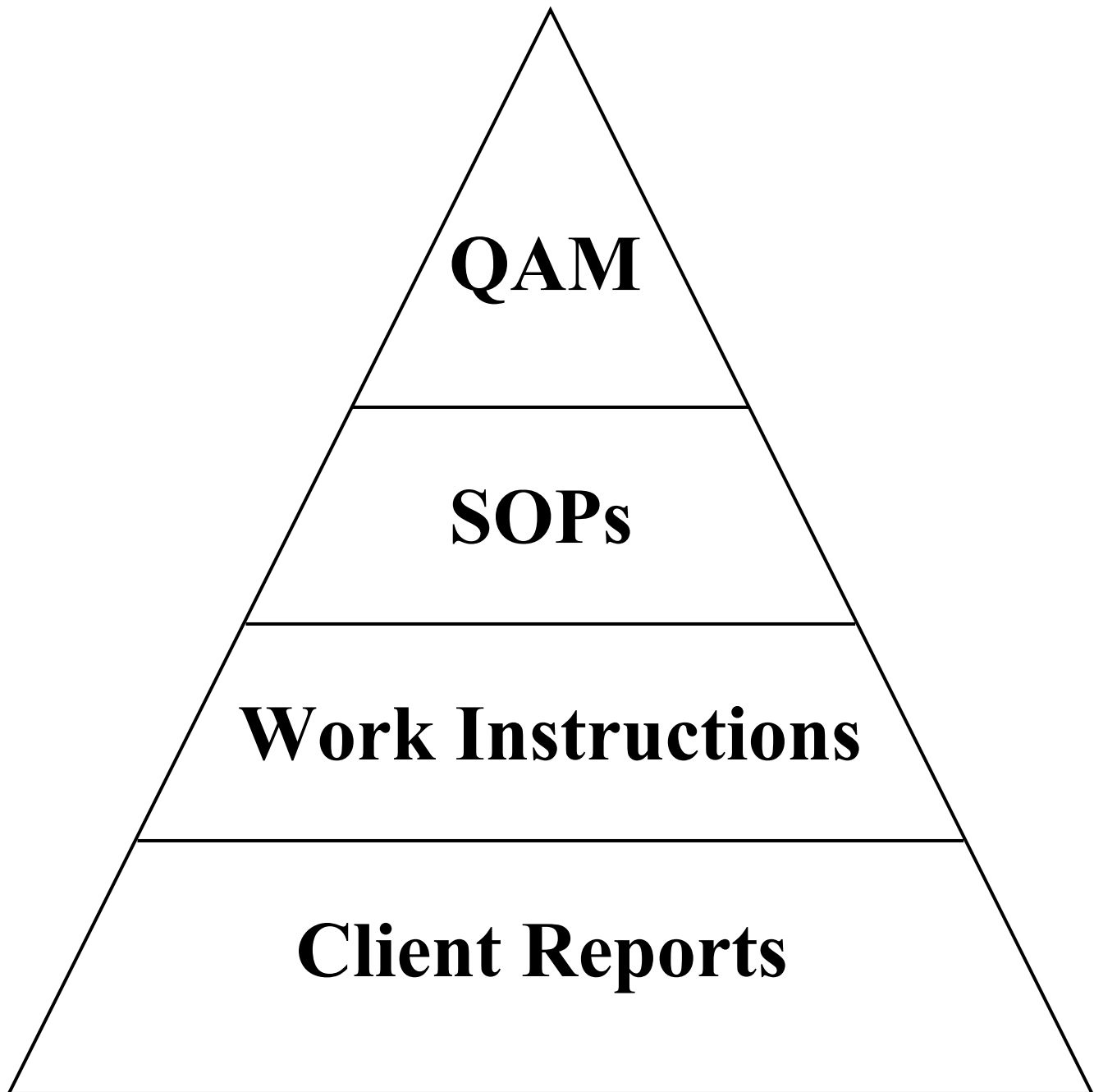
#### **4.10.2 External Audits**

##### **4.10.2.1 On-Site Audits**

Audits of the laboratory conducted by regulating agencies and client organizations are to be perceived by the laboratory staff as learning experiences and opportunities to hear suggestions from knowledgeable persons on how operations might be improved. Consequently, the laboratory staff is to be open and cooperative with external auditors. Formal follow-up using written summaries of external audits is to be carried out to ensure that any suggested improvements are thoroughly evaluated.

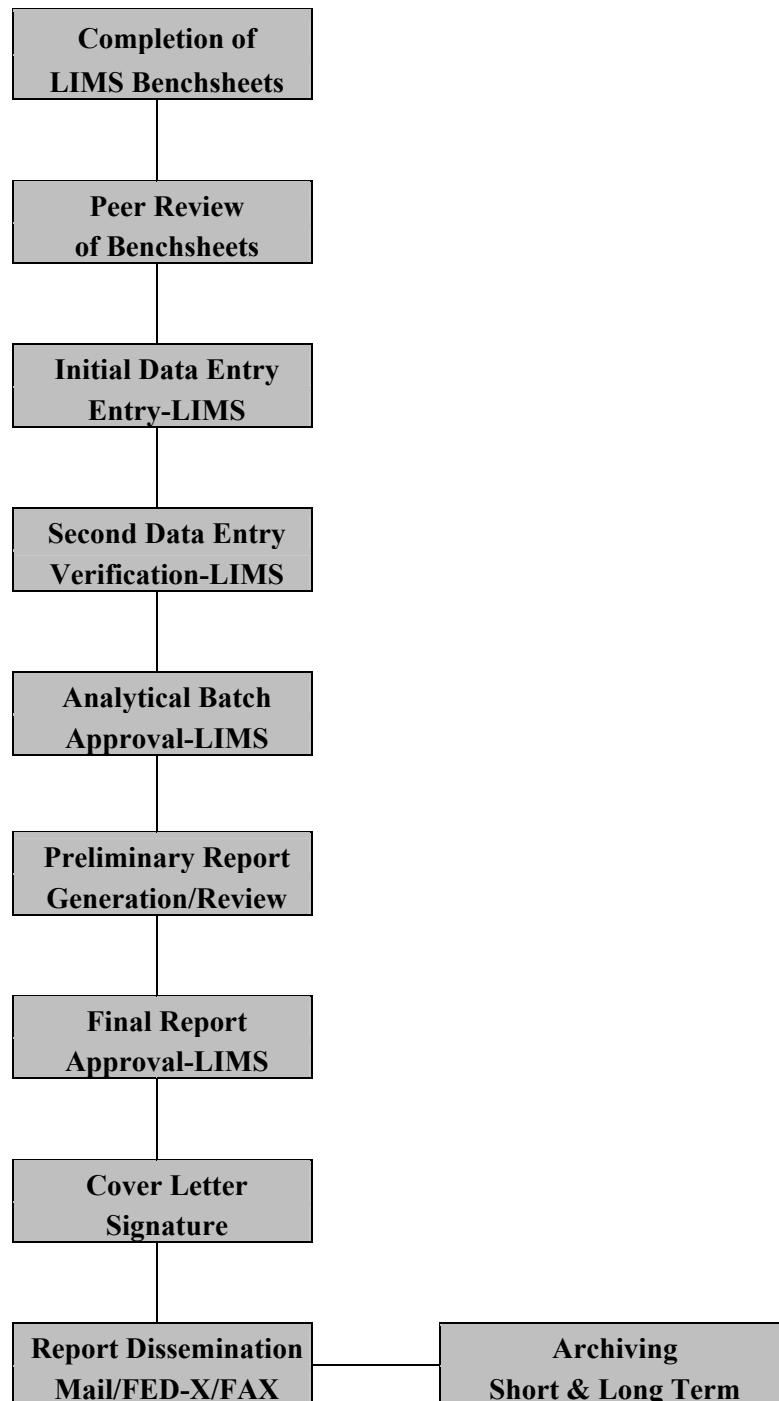


**Figure 4-1**  
**Documentation System Structure**



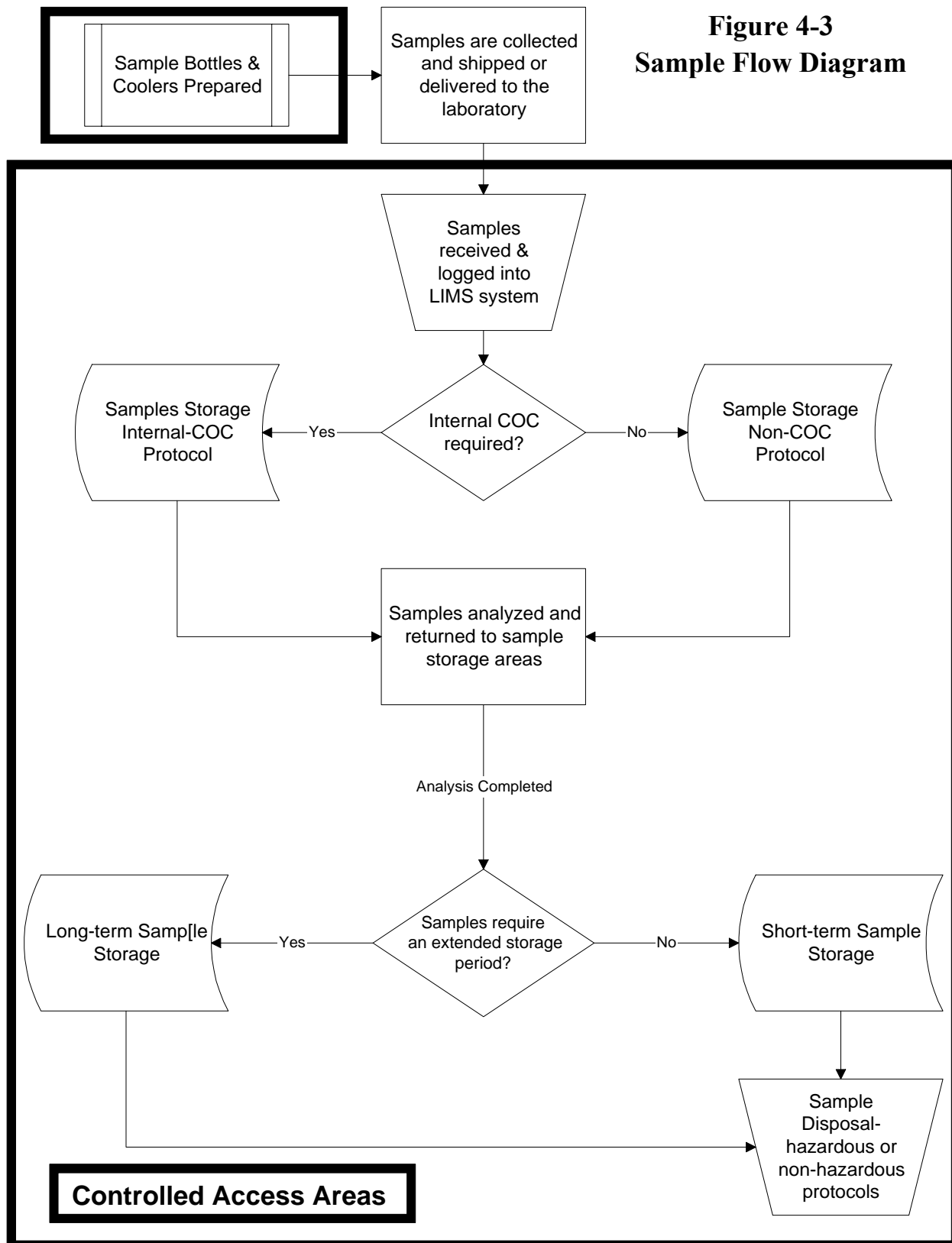


**Figure 4-2**  
**Document – Benchsheets/Client Report**  
**Flow Diagram**





**Figure 4-3  
Sample Flow Diagram**





## **5.0 REFERENCES**

- Methods for Chemical Analysis of Water and Wastes; EPA-600/4-79-020 most current revision.
- Standard Methods for the Evaluation of Water and Wastewater; Current Edition, APHA, AWWA, WPCF.
- Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories; EPA 600/4-79-019, most current revision.
- Physical and Chemical Methods for the Evaluation of Solid Waste; EPA-SW-846, most current revision.
- Guidelines Establishing Text Procedures for the Analysis of Pollutants; 40 CFR; Parts 100 to 149, Current Edition.
- Good Automated Laboratory Practices; USEPA Office of Administration and Resource Management, most current revision.



**TABLE 1**  
**Default Data Archiving Systems**

**Document Archives**

Document Description	Storage Location	Storage Duration
Laboratory benchsheets	on-site	1 year
Laboratory benchsheets	off-site	6 years
Instrument Print-Outs (raw data)	on-site	1 year
Instrument Print-Outs (raw data)	off-site	6 years
Laboratory Logs (run, maintenance, analyst)	on-site	1 year
Laboratory Logs (run, maintenance, analyst)	off-site	6 years
Client Files (reports, correspondence, invoices)	on-site	1 year
Client Files (reports, correspondence, invoices)	off-site	6 years
Proposal Files	on-site	5 years
Purchase Agreements	on-site	5 years
SOPs	on-site	5 years

**Electronic Archives**

File Description	Storage Location	Storage Duration	Storage Media
Instrument Data Files-GC/MS	on-site	1 year	Compact Disk
Instrument Data Files-GC/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-GC (Turbochrom)	on-site	1 year	Compact Disk
Instrument Data files-GC (Turbochrom) (copy)	off-site	10 years	Compact Disk
Instrument Data files-AA, ICP, ICP/MS	on-site	1 year	Compact Disk
Instrument Data files-AA, ICP, ICP/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-Auto Analyzer	on-site	1 year	Compact Disk
Instrument Data files-Auto Analyzer (copy)	off-site	10 years	Compact Disk
LIMS daily backup	on-site fire-safe	30 day rotation	DAT-Tape
SOPs	on-site	indefinitely	Compact Disk



## **TABLE 2**

### **Laboratory SOP Categories**

Trace Metals	Instrumental-General
Gas Chromatograph	Gas Chromatography/Mass Spectroscopy
Spectrophotometric Procedures	Titrimetric Procedures
Gravimetric Procedures	Electrochemical/Potentiometric Procedures
Extractions-Organic	Quality Assurance
Sales and Customer Service	Business and Accounting
Laboratory Computer Operations	Laboratory Safety and Security
Sample Receiving, Storage, & Disposal	Miscellaneous
Bottle Prep	Inorganic-General
Microbiology	
Waste Characterization	



**TABLE 3**  
**Field Equipment Calibration**

Equipment	Method Reference	Minimum # Standards Initial Calibration	Type of Curve	Frequency of Calibration	Acceptance/ Rejection Criteria Initial Calibration	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Conductivity Meter	SW-846 Method 9050	2	---	Initial	$\pm 5\%$ of Value	Daily	---
Dissolved Oxygen Meter	Standard Method 4500-O G.	---	---	Initial	$\pm 5\%$ of Value	Daily	---
Temperature Probes	Standard Method 2550 B.	---	---	Initial	$\pm 5\%$ of Value	Daily	---
pH Meter	SW-846 Method 9040	3	Linearity	Initial	Adjust slope to within $\pm 0.05$ pH units accuracy	Daily	---



**TABLE 4**  
**Instrument Calibration**

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/Rejection Criteria Continuing Calibration Verification
Mercury Cold Vapor AA	SW-846 7470/7471  EPA 245.1	5	Correlation coefficient must be $\geq 0.995$	Daily, at the beginning of every analytical batch, and when CCV fails acceptance criteria	Every calibration	90-110% recovery  95-105% recovery	Every 10 samples	90-110% recovery  90-110% recovery
ICP	SW-846 6010 EPA 200.7	3	same as above	same as above	same as above	95-105% recovery	same as above	90-110% recovery
ICP/MS	SW-846 6020 EPA 200.8	6	same as above	same as above	same as above	90-110% recovery	same as above	90-110% recovery
Ion Chromatograph	SW-846 9056 EPA 300.1	6	Correlation coefficient must be $\geq 0.995$	Every 6 months or when CCV fails	Every calibration	90-110% recovery	Every 10 samples	90-110% recovery
Konelab: Sulfate Chloride	EPA 600/4-79-020 Method 375.2 Method 325.2	10 8	same as above	Every batch	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Phenolics (Total)	SW-846 9065 EPA 420.1	5-7	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Cyanide Total and Amenable	SW-846 9012, 9014 EPA 335.1, 335.3, 335.4	7	same as above	same as above	same as above	90-110% recovery	Every 10 samples	90-110% recovery



**TABLE 4**  
**Instrument Calibration**

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/Rejection Criteria Continuing Calibration Verification
TOC Analyzer-TOC	EPA 415.1	5	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
GC-PID/ ELCD	SW-846 8021	5 for linear 6 for quadratic	≤20% RSD use average RF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; ±20% for compounds that boil below 30° C (Bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, and vinyl chloride)
	EPA 601/602	3	<10% RSD use average RF or regression, ≥10% must use regression	As needed when CCV fails method Table 2 criteria				Method Table 2 criteria
GC-FID	SW-846 8015	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration



**TABLE 4**  
**Instrument Calibration**

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/Rejection Criteria Continuing Calibration Verification
GC-ECD	SW-846 8081 SW-846 8151 SW-846 8082 SW-846 8121  EPA 608 EPA 612	5 for linear 6 for quadratic  3	≤20% RSD use average CF or regression, >20% must use regression  <10% RSD use average CF or regression, ≥10% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; breakdown criteria: DDT <15% Endrin <15%, <20% total
GC-HPLC	SW-846 8310	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration



**TABLE 4**  
**Instrument Calibration**

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/Rejection Criteria Continuing Calibration Verification
GC/MS-Volatiles	SW-846 8260	5 for linear 6 for quadratic	CCCs – %RSD $\leq 30\%$ 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene ethyl benzene, vinyl chloride, all other target analytes $\leq 15\%$ use average RF for quantitation, otherwise regression SPCCs – average RF $\geq 0.10$ for chloromethane, 1,1-dichloroethane and bromoform; $\geq 0.30$ for 1,1,2,2-tetrachloroethene and chlorobenzene	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8260: CCCs – % Difference or drift $\leq 20\%$ , all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 624	3	$< 35\%$ RSD for all compounds use average RF, otherwise use regression				24 hours	Recovery of all analytes must meet recoveries specified in Table 5



**TABLE 4**  
**Instrument Calibration**

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/Rejection Criteria Continuing Calibration Verification
GC/MS-Semi-volatiles	SW-846-8270	5 for linear 6 for quadratic	CCCs – %RSD $\leq$ 30% acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-diphenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, 2,4,6-trichlorophenol, all other target analytes $\leq$ 15% use average RF for quantitation, otherwise regression SPCCs – average RF $\geq$ 0.05 N-nitrosodi-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8270: CCCs % Difference or drift $\leq$ 20%; all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 625	3	$<$ 35% RSD for all compounds use average RF, otherwise use regression				24 hours	80-120% recovery



**TABLE 5**  
**Quality Control Sample Types**

<b>Blank Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Method Preparation Blank	MPB	This blank has been carried through the entire analytical process including any pretreatment procedures. The MPB will monitor any contaminants that may affect the sample results. General acceptance limits for the MPB are less than the test reporting Limit. If contamination is detected in the MPB above the reporting limit, all samples with analyte concentrations within 10x that found in the MPB must be flagged for re-extraction or digestion. If it is not possible to re-prep the samples then all analyses for that batch must be qualified.	One per analytical batch



**TABLE 5**  
**Quality Control Sample Types**

<b>Blank Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Continuing Calibration Blank	CCB	<p>The continuing calibration blank is a reagent blank that is analyzed as a sample, generally after 10 samples have been tested. The CCB must be run prior to re-zeroing an instrument, unless this practice was performed for each previous sample. The CCB will verify whether significant instrument drift has occurred during the analytical run near the test method detection limit. General acceptance limits are <math>\pm</math> the test reporting limit. If the CCB falls outside the acceptance limits, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analyses are completed, 10 samples before and after the unacceptable CCB must be reanalyzed, i.e., all sample results must be encased in acceptable CCB. The reanalysis must also include the ICB and ICV QC samples.</p>	Every ten samples/or as specified in the analytical method.



**TABLE 5**  
**Quality Control Sample Types**

<b>Blank Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Field Trip Blank	FTB	<p>These are used with VOA vials where there is the possibility that organic contaminants may diffuse through the PTFE-faced silicone rubber septum of the sample vial.</p> <p>A field trip blank vial filled with organic-free water accompanies the sample containers to and from a client location, at the discretion of the client, may be analyzed along with the samples.</p>	One per sample shipping container
Storage Blank	STB	<p>Reagent-grade water (40 mL aliquot) is stored with samples in a client set.</p> <p>Per the discretion of the client, it may be analyzed after all samples in that set are analyzed. The purpose is to determine the level of contamination acquired during storage.</p>	One per sample storage refrigerator or client sample set (if required)



**TABLE 5**  
**Quality Control Sample Types**

<b>Control Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Laboratory Fortified Blank or Blank Spike	LFB or BS	This is a fortified method preparation blank in which an aliquot of de-ionized water has been spiked with a known amount of a stock reference standard or spiking solution. A blank spike is required for each digestion or distillation batch. The purpose of the blank spike is to verify the analyst's spiking procedure and assure that any matrix interference shown by the spike and spike duplicate is really matrix induced.	One per analytical batch or as specified in the analytical method



**TABLE 5**  
**Quality Control Sample Types**

<b>Control Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Second-Source Calibration Verification	SCV	The SCV is identical to the CCV with the exception it must be made from a source dissimilar to that used to prepare the initial calibration curve. The purpose of the SCV is to validate the accuracy both the calibration standards, and the initial calibration curve. Unless otherwise specified by the method, recovery limits for this QC type are typically 80-120%. Sample analysis may not begin prior to the analysis of a successful SCV.	One with every initial calibration



**TABLE 5**  
**Quality Control Sample Types**

<b>Control Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Continuing Calibration Verification	CCV	<p>The continuing calibration verification standard is generally the standard used as the midpoint of the initial calibration curve.</p> <p>The standard is analyzed and quantitated in the same manner as a sample. The CCV will reveal any significant instrument drift. Acceptance limits for this QC type are <math>\pm 10\%</math>, or as stated in the method. If the CCV falls outside the acceptance window, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analysis is complete, all samples run after the last acceptable CCV must be reanalyzed, i.e. all samples must be bracketed by an acceptable CCV.</p>	Every 10 samples or as specified in the analytical method



**TABLE 5**  
**Quality Control Sample Types**

<b>Control Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Detection Limit	CRDL	A standard which contains the minimum level of detection acceptable under a contract Statement of Work must be analyzed for particular contract sample sets to demonstrate that detection limit can be met.	One per analytical batch for certain contract sample sets and methods only.
Sample Duplicate	DUP	The sample duplicate is a replicate analysis of a particular sample that has been analyzed previously during the sample analytical batch. The purpose of the duplicate is to monitor precision within the analytical process.	Every 10 samples for each matrix type



**TABLE 5**  
**Quality Control Sample Types**

<b>Control Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Sample Matrix Spike	SPK	The sample matrix spike is an aliquot of a sample that has been spiked with a known amount of a stock reference standard or spiking solution. A the purpose of the SPK is to monitor sample matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type, or as specified in the analytical method



**TABLE 5**  
**Quality Control Sample Types**

<b>Matrix QC Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Matrix Spike Duplicate	MSD	A matrix spike duplicate is an aliquot of the same sample used for the matrix spike (SPK). A spike duplicate is required for each matrix type within a digestion or distillation batch. A spike duplicate analysis may be required on a non-distilled or non-digested sample if the spike has indicated a matrix interference. The purpose of this duplicate spike is to confirm any matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type or as specified in the analytical method



**TABLE 5**  
**Quality Control Sample Types**

<b>Matrix QC Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Field Duplicate	FDUP	<p>This may be required to evaluate the uniformity of samples and sampling techniques at a field location.</p> <p>Acceptance limits for this QC type are based on established confidence limits, with generally two levels or ranges. The first range extends from the test reporting limit to 10x the test reporting limit. The second range encompasses any values higher than 10x the MDL.</p>	As required on a project basis
Post-Digestion Spike	PDS	<p>The post-digestion spike may be required, on a project basis, when a matrix precludes the use of pre-digestion spike.</p>	One per analytical batch when required by project



**TABLE 5**  
**Quality Control Sample Types**

<b>Matrix QC Type</b>	<b>Abbreviation</b>	<b>Description</b>	<b>Frequency of Use</b>
Surrogate Spike	SUR	For almost all organic analyses, the analytical method requires surrogate compounds to be added to every blank, sample, matrix spike, matrix spike duplicate, and standard. Surrogate compounds are used to measure analytical efficiency by measuring percent recovery from the known value. They are generally brominated, fluorinated, or isotopically labeled compounds not typically detected in environmental samples.	Every QC and per batch for semi-volatile, volatile, pesticide, PCB analysis
Internal Standard	IST	These are compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), at a known concentration, prior to analysis. Internal standards are used as the basis of quantitation of the target compounds.	Every QC and client sample per batch for volatiles and semi-volatiles



# Appendix A



## CHEMIST I

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### **General Description**

Under direct supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

### **Educational/Background Requirements**

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or
- BS degree in Chemistry or a related field of science.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Chemist I.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Perform all other activities deemed necessary to management.



## CHEMIST II

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### **General Description**

Under ***general*** supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

### **Educational/Background Requirements**

- Associates degree and **5** or more years of experience in an ***applicable discipline***; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline***; or
- ***MS degree in Chemistry or a related field of science.***

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Chemist II.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other chemists and technicians with their professional development.***



- *Act as company advocate by setting a positive example in work habits and attitude to other staff members.*
- *Demonstrate ability to work independently with minimal errors.*
- *Capable of conducting peer review on routine data packages.*
- *Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.*
- Perform all other activities deemed necessary to management.



## CHEMIST III

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### **General Description**

Under *minimal* supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties. *Eligible for consideration of group leader status.*

### **Educational/Background Requirements**

- Associates degree and *7 or more years of experience in an applicable discipline*; or
- BS degree in Chemistry or a related field of science and *4* or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science *and 2 or more years of experience in an applicable discipline.*

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Chemist III.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/*non-routine* maintenance *and troubleshooting* of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other chemists and technicians with their professional development.



- Act as company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate *increased* ability to work independently with minimal errors.
- Capable of conducting peer review on routine *and non-routine* data packages. *Has demonstrated knowledge to perform final data review and approval on LIMS.*
- Possess *an above average* level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- *Assist in the development and maintenance of laboratory SOPs.*
- Perform all other activities deemed necessary to management.



## CHEMIST IV

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### **General Description**

Under minimal supervision of the area manager and/or *the technical director*, conducts **complex** analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- Associates degree and **10** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **7** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and **4** or more years of experience in an applicable discipline; or
- *Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting.*

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Chemist IV.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation *of, and assisting other chemists in*, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.



- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other chemists and technicians with their professional development *and in the integration of new methods and technologies.*
- Act as company advocate by setting a positive example in work habits and attitude to other staff members, *prospective employees, existing and perspective clientele, and the general public.*
- Demonstrate *superior* ability to work independently with minimal errors.
- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.
- Possess *a superior* level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- *Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.*
- *When appropriate, work with the technical director to develop new methods and technologies.*
- *Develop, review, and update laboratory SOPs as necessary.*
- Perform all other activities deemed necessary to management.



## CHEMIST V

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### **General Description**

Under minimal supervision of the area manager and/or the technical director, conducts complex analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status. *May work directly with the technical director to develop new methods and technologies for the laboratory.*

### **Educational/Background Requirements**

- Associates degree and **13** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **10** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and **6** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and **2 or more years of** experience in an environmental or related laboratory setting.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Chemist V.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, *and serving as the primary reference for*, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.



- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.
- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.
- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.
- ***Responsible for the study and implementation of*** new methods and technologies.
- Develop, review, and update existing laboratory SOPs as necessary, ***write new SOPs as required to reflect advancements in methods and technologies.***
- ***Work with management team to plan for future equipment acquisitions.***
- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.***
- Perform all other activities deemed necessary to management.





## SENIOR CHEMIST

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### **General Description**

*Working independently or* under minimal supervision of, *an* area manager, technical director, *or the laboratory president*, conducts *or supervises analysis of complex non-routine projects* to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- BS degree in Chemistry or a related field of science and **15** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and **10** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and **7** or more years of experience in an environmental or related laboratory setting.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Senior Chemist.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.



- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.
- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.
- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.
- Responsible for the study and implementation of new methods and technologies.
- Develop, review, and update existing laboratory SOPs as necessary, write new SOPs as required to reflect advancements in methods and technologies.
- Work with management team to plan for future equipment acquisitions.
- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.
- Perform all other activities deemed necessary to management.





## PROJECT CHEMIST I

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### **General Description**

Under direct supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

### **Educational/Background Requirements**

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or
- BS degree in Chemistry or a related field of science.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Project Chemist I.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness.
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare proposal outlines for existing clients.
- Perform all other activities deemed necessary to management.





## PROJECT CHEMIST II

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### **General Description**

Under **general** supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

### **Educational/Background Requirements**

- Associates degree and **5** or more years of experience in an ***applicable discipline***; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline***; or
- ***MS degree in Chemistry or a related field of science.***

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Project Chemist II.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness.
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare proposal outlines for existing ***and new*** clients.
- ***Assist other project chemists and technicians with their professional development.***



- *Act as a company advocate by setting a positive example in work habits and attitude to other staff members.*
- *Demonstrate ability to work independently with minimal errors.*
- *Posses the minimum level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.*
- Perform all other activities deemed necessary to management.



## PROJECT CHEMIST III

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### **General Description**

Under *minimal* supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs. ***Eligible for consideration of group leader status.***

### **Educational/Background Requirements**

- Associates degree and **7** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **4** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Project Chemist III.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. ***Assist with the preparation, archiving, and delivery of a CLP or "CLP Like" deliverables package.***
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.



- *Prepare and/or coordinate the preparation of proposals for existing and new clients under direct supervision of the client services manager, sales manager, or laboratory president.*
- Assist other project chemists and technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate **increased** ability to work independently with minimal errors.
- Posses **an above average** level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- *Demonstrate ability to improve productivity as shown by an increase in project workload and throughput.*
- *Provide data interpretation services to clients.*
- *Assist in the development and maintenance of laboratory SOPs.*
- Perform all other activities deemed necessary to management.



## PROJECT CHEMIST IV

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### **General Description**

Under minimal supervision of the client services manager and/or *the sales manager*, acts as the primary interface with the client to assure laboratory services are meeting client needs. *May work directly with the sales manager to develop increased business from existing clients.* Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- Associates degree and **10** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **7** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and **4** or more years of experience in an applicable discipline; or
- *Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting.*

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Project Chemist IV.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, *and assist other project chemists with*, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. *Coordinate* the preparation, archiving, and delivery of CLP or “CLP Like” deliverables packages.



- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare and/or coordinate the preparation of proposals for existing and new clients under *minimum* supervision of the client services manager, sales manager, or laboratory president.
- Assist other project chemists and technicians with their professional development *and in the integration of new methods and technologies*.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, *prospective employees, existing and perspective clientele, and the general public*.
- Demonstrate *superior* ability to work independently with minimal errors.
- Posses *a superior* level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput *as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs*.
- Provide data interpretation services to clients.
- *Develop, review, and update laboratory SOPs as necessary.*
- *When appropriate, work with sales manager to develop additional business from existing clients.*
- Perform all other activities deemed necessary to management.



## PROJECT CHEMIST V

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### **General Description**

Under minimal supervision of the client services manager and/or the sales manager, acts as the primary interface with the client to assure laboratory services are meeting client needs. **Works** directly with the sales manager to **establish relationships with new clients as well as increase** business from existing clients. Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- Associates degree and **13** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **10** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and **6** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and **2 or more years of** experience in an environmental or related laboratory setting.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Project Chemist V.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or “CLP Like” deliverables packages.



- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. ***Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.***
- Assist other project chemists and technicians with their professional development and ***serve as the primary reference for*** the integration of new methods and technologies.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs. ***Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.***
- Provide data interpretation services to clients. ***Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.***
- Develop, review, and update laboratory SOPs as necessary. ***Write new SOPs as required to reflect advancements in procedures or technologies.***
- ***Routinely*** work with sales manager to develop additional business from existing clients ***and new clients.***
- ***Responsible for the study and implementation of new procedures and technologies.***
- ***Work with management team to plan for future equipment and software acquisitions.***
- ***Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.***
- Perform all other activities deemed necessary to management.





## SENIOR PROJECT CHEMIST

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### **General Description**

*Working independently or* under minimal supervision of the client services manager and/or the sales manager, *or laboratory president*, acts as the primary interface with the client to assure laboratory services are meeting client needs. Works directly with the sales manager to establish relationships with new clients as well as increase business from existing clients. *Works directly with the laboratory president to develop the laboratory portion of QAPPs, work plans, and other technical documents.* Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- BS degree in Chemistry or a related field of science and **15** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and **10** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and **7** or more years of experience in an environmental or related laboratory setting.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Senior Project Chemist.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or "CLP Like" deliverables packages.



- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.
- Assist other project chemists and technicians with their professional development and serve as the primary reference for the integration of new methods and technologies.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs. Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.
- Provide data interpretation services to clients. Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.
- Develop, review, and update laboratory SOPs as necessary. Write new SOPs as required to reflect advancements in procedures or technologies.
- Routinely work with sales manager to develop additional business from existing clients and new clients.
- Responsible for the study and implementation of new procedures and technologies.
- Work with management team to plan for future equipment and software acquisitions.
- Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.
- Perform all other activities deemed necessary to management.





## TECHNICIAN I

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### **General Description**

Under direct supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

### **Educational/Background Requirements**

- High school diploma or equivalent.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Technician I.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Perform all other activities deemed necessary to management.



## TECHNICIAN II

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### **General Description**

Under **general** supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

### **Educational/Background Requirements**

- High school diploma or equivalent ***and 2 or more years of experience in an applicable discipline;*** or
- ***Associates degree and 1 or more years of experience in an applicable discipline;*** or
- ***BS degree in Chemistry or a related field of science.***

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Technician II.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other technicians with their professional development.***
- ***Act as a company advocate by setting a positive example in work habits and attitude to other staff members.***



- *Demonstrate ability to work independently with minimal errors.*
- *Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.*
- Perform all other activities deemed necessary to management.



## TECHNICIAN III

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### **General Description**

Under *minimal* supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory. *Eligible for consideration of group leader status.*

### **Educational/Background Requirements**

- High school diploma or equivalent and **4** or more years of experience in an applicable discipline; or
- Associates degree and **3** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science *and 2 or more years of experience in an applicable discipline.*
- *MS degree in Chemistry or a related field of science.*

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Technician III.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/*non-routine* maintenance *and troubleshooting* of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.



- Assist other technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate *increased* ability to work independently with minimal errors.
- Possess *an above average* level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- *Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput.*
- *Assist in the development and maintenance of laboratory SOPs.*
- Perform all other activities deemed necessary to management.





## TECHNICIAN IV

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### **General Description**

Under minimal supervision of the area manager and/or *the technical director*, performs **complex** tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- High school diploma or equivalent and **7** or more years of experience in an applicable discipline; or
- Associates degree and **5** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **4** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science *and 2 or more years of experience in an applicable discipline.*

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Technician IV.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation *of, and assisting other technicians in*, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.



- Assist other technicians with their professional development *and in the integration of new procedures and technologies.*
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, *prospective employees, existing and prospective clientele, and the general public.*
- Demonstrate *superior* ability to work independently with minimal errors.
- Possess *a superior* level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, *addition of new procedures/technologies and/or operation of additional equipment/instruments.*
- *When appropriate, work with the technical director, laboratory president, or sales manager to develop new procedures and technologies.*
- *Develop, review, and update laboratory SOPs as necessary.*
- Perform all other activities deemed necessary to management.



## TECHNICIAN V

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### **General Description**

Under minimal supervision of the area manager and/or the technical director, performs complex tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status. ***May work directly with the technical director, laboratory president, or sales manager to develop methods, procedures, and technologies for the laboratory.***

### **Educational/Background Requirements**

- High school diploma or equivalent and **10** or more years of experience in an applicable discipline; or
- Associates degree and **8** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **6** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and **4** or more years of experience in an applicable discipline.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Technician V.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, ***and serving as the primary reference for***, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.



- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other technicians with their professional development and in the integration of new procedures and technologies.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.
- ***Responsible for the study and implementation of*** new procedures and technologies.
- Develop, review, and update laboratory SOPs as necessary, ***write new SOPs as required to reflect advancement in procedures and technologies.***
- ***Work with management team to plan for future equipment acquisitions.***
- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.***
- Perform all other activities deemed necessary to management.





## SENIOR TECHNICIAN

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### **General Description**

*Working independently or* under minimal supervision of, *an* area manager, technical director, *or the laboratory president*, performs *or supervises tasks related to complex non-routine projects* necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

### **Educational/Background Requirements**

- High school diploma or equivalent and **15** or more years of experience in an applicable discipline; or
- Associates degree and **13** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and **10** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and **7** or more years of experience in an applicable discipline.

### **Minimum Required Skills and Responsibilities**

The following are the minimum skills and responsibilities required of a Senior Technician.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.



- Assist other technicians with their professional development and in the integration of new procedures and technologies.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.
- Demonstrate superior ability to work independently with minimal errors.
- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.
- Responsible for the study and implementation of new procedures and technologies.
- Develop, review, and update laboratory SOPs as necessary, write new SOPs as required to reflect advancement in procedures and technologies.
- Work with management team to plan for future equipment acquisitions.
- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.
- Perform all other activities deemed necessary to management.



## **GROUP LEADER**

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### **General Description**

In addition to the duties associated with the current chemist level, a group leader also takes on administrative responsibilities involved with the operation of the laboratory area.

### **Educational/Background Requirements**

- Minimum of those specified with a Chemist III.

### **Minimum Required Skills and Responsibilities**

Consistent with current Chemist Level, with additional or increased emphasis on the following requirements.

- Act as the area manager when the area manager is absent, filling such duties as supervision of employees and review and approval of data.
- Act as an additional source of information for management and others regarding laboratory area analysis capabilities.
- Responsible for the scheduling of work and the monitoring of workload for such items as hold times and due dates.
- Provide leadership, guidance, and training to other laboratory personnel on methods, equipment, and quality control.
- Develop, review and update laboratory SOPs as necessary.
- Assure that new methods, policies, and procedures are integrated into the laboratory area.
- Assume a primary responsibility for verifying that sample analyses are adhering to all method and laboratory specified quality assurance parameters.



# Appendix B





## Inorganic Analyses

Parameter	Reference Citation
ACIDITY AS CaCO <sub>3</sub>	SDM 2310 B
ALKALINITY, BICARBONATE	SDM 2320 B
ALKALINITY, CARBONATE	SDM 2320 B
ALKALINITY, HYDROXIDE	SDM 2320 B
ALKALINITY, PHENOLPHTHALEIN	SDM 2320 B
ALKALINITY, TOTAL	SDM 2320 B
BOD, (5-DAY)	SDM 5210 B
BOD, (5-DAY), DISSOLVED	SDM 5210 B
BOD, CARBONACEOUS (5-DAY)	SDM 5210 B
BROMIDE	USEPA 9056, ASTM D1246-88
CARBON DIOXIDE	SDM 4500-CO <sub>2</sub> C
CARBON, DISSOLVED ORGANIC	USEPA 9060, SDM 5310 D
CARBON, PURGEABLE ORGANIC	USEPA 9060
CARBON, TOTAL INORGANIC	USEPA 9060
CARBON, TOTAL ORGANIC	USEPA 9060, MSA 29.3.5.2, SDM 5310 D
CARBON, ORGANIC (NON-PURGE)	USEPA 9060
CATION EXCHANGE CAPACITY	USEPA-9081
CHEMICAL OXYGEN DEMAND	SDM 5220 D
CHLORIDE	SDM 4500-Cl B, USEPA 300.0/9056
CHLORINE, TOTAL RESIDUAL	HACH-8167
CHROMIUM, HEXAVALENT	SDM 3500-Cr D/USEPA 7196A
COLIFORM, FECAL	SDM 9222 D
COLIFORM, TOTAL	SDM 9223 B
COLOR (APPARENT)	SDM 2120 B
CONDUCTIVITY @ 25°C	USEPA-120.1/9050A, SDM 2510 B
CORROSION TOWARD STEEL	USEPA-1110
CYANIDE, AMENABLE	USEPA-9012A, SDM 4500-CN G
CYANIDE, FREE	USEPA-9014
CYANIDE, WEAK ACID DIS.	SDM-4500-CN I
CYANIDE, TOTAL	USEPA-335.4/9012A
DENSITY	SDM 2710 F
EXTRACTABLE ORGANIC HALIDES-EOX	USEPA-9023
FLUORIDE	USEPA-300.0/9056, SDM 4500-F C
FORMALDEHYDE	USEPA-8315A
GROUNDWATER DEPTH	USGS
GROUNDWATER LEVEL	USGS
HARDNESS, TOTAL	SDM 2340 C
HEM; OIL & GREASE	USEPA-1664/9070A/9071B
HETEROTROPHIC PLATE COUNT	SDM 9215 B
IGNITABILITY, SETAFLASH CLOSED-CUP	USEPA-1020A
IRON, FERRIC BY CALCULATION	SDM 3500-Fe D
IRON, FERROUS	SDM 3500-Fe D
NITROCELLULOSE	USARMY BR&D Lab
NITROGEN, AMMONIA	SDM 4500-NH <sub>3</sub> G
NITROGEN, INORGANIC (NH <sub>4</sub> )	SDM 4500-NH <sub>3</sub> G
NITROGEN, INORGANIC (NO <sub>3</sub> +NO <sub>2</sub> )	USEPA-353.2, SDM 4500-NO <sub>3</sub> F
NITROGEN, INORGANIC	USEPA-350.1 + 353.2
NITROGEN, NITRATE	USEPA-300.0/353.2/9056, SDM 4500-NO <sub>3</sub> F





## Inorganic Analyses

Parameter	Reference Citation
NITROGEN, NITRATE+NITRITE	USEPA-353.2, SDM 4500-NO <sub>3</sub> F
NITROGEN, NITRITE	USEPA-300.0/353.2/9056, SDM 4500-NO <sub>2</sub> B
NITROGEN, ORG. (NH <sub>4</sub> )	USEPA-350.1
NITROGEN, ORGANIC	USEPA-351.2
NITROGEN, TOTAL KJELDAHL	USEPA-351.2
ODOR	SDM 2150 B
OXYGEN, DISSOLVED	SDM 4500-O G
PAINT FILTER LIQUIDS TEST	USEPA-9095
PERCENT ASH	USEPA-160.4
PERCENT MOISTURE	SDM 2540 B
PERCENT SOLIDS	SDM 2540 B
PERCENT VOLATILE SOLIDS	USEPA-160.4, SDM 2540 G
PH	USEPA-150.1/9040B/9045C
PHENOLICS, TOTAL	USEPA-420.1/B17420.2/9066
PHOSPHORUS, ORTHO	SDM 4500-P E
PHOSPHORUS, TOTAL	USEPA-365.1, SDM 4500-P F
PHOSPHORUS, TOTAL-SOLUBLE	USEPA-365.1, SDM 4500-P F
RESIDUE, DISSOLVED @ 180C	SDM 2540 C
RESIDUE, DISSOLVED-VOL.	USEPA-160.4
RESIDUE, SUSPENDED	SDM 2540 D
RESIDUE, SUSPENDED-VOL.	USEPA-160.4
RESIDUE, TOTAL	SDM 2540 B
RESIDUE, TOTAL-VOLATILE	USEPA-160.4, SDM 2540 G
SGT-HEM; NON-POLAR MATERIAL	USEPA-1664/9070A/9071B
SILICA, DISSOLVED	SDM 4500-SiO <sub>2</sub> D
SODIUM HEXAMETAPHOSPHATE	USEPA-365.1
SPECIFIC GRAVITY	ASTM-D 1429-79, SDM 2710 F
STATIC WATER LEVEL	USGS
SULFATE	USEPA-300.0/375.2/9056/9038, SDM 4500-SO <sub>4</sub> F
SULFIDE	USEPA-9034, SDM 4500-S <sub>2</sub> F
SULFIDES, ACID VOLATILE	ET&C VOL 12
SULFITE	SDM 4500-SO <sub>3</sub> B
SURFACTANTS, MBAS	SDM 5540 C
TEMPERATURE	SDM 2550 B
THIOCYANATE	SDM 4500-CN M
TOTAL ORGANIC HALIDES	USEPA-9020B/9023
TURBIDITY	SDM 2130 B





## Metals Analyses

Parameter	Reference Citation
ALUMINUM, ICP	USEPA-200.7/6010B
ANTIMONY, ICP	USEPA-200.7/6010B
ANTIMONY, MS	USEPA-200.8/6020
ARSENIC, ICP	USEPA-200.7/6010B
ARSENIC, MS	USEPA-200.8/6020
BARIUM, ICP	USEPA-200.7/6010B
BARIUM, MS	USEPA-200.8/6020
BERYLLIUM, ICP	USEPA-200.7/6010B
BERYLLIUM, MS	USEPA-200.8/6020
BORON, ICP	USEPA-200.7/6010B
BORON, MS	USEPA-200.8/6020
CADMIUM, ICP	USEPA-200.7/6010B
CADMIUM, MS	USEPA-200.8/6020
CALCIUM AS CaCO <sub>3</sub>	USEPA-200.7/6010B
CALCIUM, ICP	USEPA-200.7/6010B
CHROMIUM, ICP	USEPA-200.7/6010B
CHROMIUM, MS	USEPA-200.8/6020
COBALT, ICP	USEPA-200.7/6010B
COBALT, MS	USEPA-200.8/6020
COPPER, ICP	USEPA-200.7/6010B
COPPER, MS	USEPA-200.8/6020
HARDNESS BY CALCULATION, ICP	USEPA-200.7/6010B
IRON, ICP	USEPA-200.7/6010B
LEAD, ICP	USEPA-200.7/6010B
LEAD, MS	USEPA-200.8/6020
LITHIUM, ICP	USEPA-200.7/6010B
MAGNESIUM AS CaCO <sub>3</sub> , ICP	USEPA-200.7/6010B
MAGNESIUM, ICP	USEPA-200.7/6010B
MANGANESE, ICP	USEPA-200.7/6010B
MANGANESE, MS	USEPA-200.8/6020
MERCURY, COLD VAPOR	USEPA-245.1/7470A/7471A
MOLYBDENUM, ICP	USEPA-200.7/6010B
MOLYBDENUM, MS	USEPA-200.8/6020
NICKEL, ICP	USEPA-200.7/6010B
NICKEL, MS	USEPA-200.8/6020
PHOSPHORUS, ICP	USEPA-200.7/6010B
POTASSIUM, ICP	USEPA-200.7/6010B
SELENIUM, ICP	USEPA-200.7/6010B
SELENIUM, MS	USEPA-200.8/6020
SILICON, ICP	USEPA-200.7/6010B
SILVER, ICP	USEPA-200.7/6010B
SILVER, MS	USEPA-200.8/6020
SODIUM, ICP	USEPA-200.7/6010B
STRONTIUM, TOTAL	USEPA-200.7/6010B
THALLIUM, ICP	USEPA-200.7/6010B
THALLIUM, MS	USEPA-200.8/6020
TIN, ICP	USEPA-200.7/6010B
TIN, MS	USEPA-200.8/6020
TITANIUM, ICP	USEPA-200.7/6010B





### Metals Analyses

Parameter	Reference Citation
VANADIUM, ICP	USEPA-200.7/6010B
VANADIUM, MS	USEPA-200.8/6020
ZINC, ICP	USEPA-200.7/6010B
ZINC, MS	USEPA-200.8/6020





## Semi-Volatile Organic Analyses

Parameter	Reference Citation
HPLC ACRYLAMIDE	EPA-8316
GC ORGANOCHLORINE PESTICIDES	USEPA-608/8081A
GC METHOXYCHLOR	USEPA-608.2
HPLC POLYNUCLEAR AROMATIC HYDROCARBONS	USEPA-610/8310
GC/MS BASE/NEUTRAL/ACIDS	USEPA-625/8270C
GC ANALYSIS OF 1,2-DIBROMOMETHANE/ 1,2-DIBROMO-3-CHLOROPROPANE/ 1,2,3-TRICHLOROPROPANE BY MICROEXTRACTION	USEPA-8011
GC DIESEL RANGE ORGANICS	USEPA-8015B, CALIFORNIA LUFT METHOD, WISCONSIN METHOD PUBL-SW-141
GC GLYCOLS	USEPA-8015B
GC POLYCHLORINATED BIPHENYLS	USEPA-8082
GC CHLORINATED HYDROCARBONS	USEPA-8121
GC HERBICIDES	USEPA-8151A
HPLC ALDEHYDES	USEPA-8315A
HPLC NITROAROMATICS AND NITRAMINES	USEPA-8330
HPLC NITROGLYCERINE	USEPA-8332





### **Volatile Organic Analyses**

<b>Parameter</b>	<b>Reference Citation</b>
GC GASOLINE RANGE ORGANICS	USEPA-8015B, CALIFORNIA DHS LUFT, IOWA-PA1, WISCONSIN METHOD PUBL-SW-140
GC AIR ANALYSIS	40CFR METHOD 18
GC DISSOLVED HEADSPACE ANALYSIS OF METHANE/ETHANE/ETHYLENE	RSK-175
GC ALCOHOLS	USEPA-8015B
GC VOLATILE ORGANICS	USEPA-601/602/8021B
GC/MS VOLATILE ORGANICS	USEPA-524.2/624/8260B



# Appendix C





## Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
190	Administration	Sonicator - Fisher	F550,# F1520			
200	Administration	Sonicator - Fisher	F550,# F1309			
209	Administration	Mettler PC4400 Toploading Balance				
212	Administration	Mettler AB204 Analytical Balance				
215	Administration	Denver Instrument P-4002 Toploading Balance				
225	Administration	Dionex Accelerated Solvent Extractor	Model ASE 300	8/2003	3070313	New
300	Administration	Fisher Ultrasonic Cleaner	FS110			
301	Administration	Fisher Heated Ultrasonic Cleaner	FS21H			
307	Administration	Denver Instruments Top Loading Balance	Model P-2002	1/2007	P2K2126010	New
317	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2005	05G1614AM	New
318	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2004	04C2296AE	New
319	Client Services	HACH Turbidimeter	Model 2100P		30300030404	New
320	Client Services	Fisher Accumet Multi-Parameter Meter	Model AP84		274436	New
100	Wet Chemistry	Orion pH/ISE Meter	8102			
120	Wet Chemistry	Ultraviolet Spectrophotometer Shimadzu	1601			
161	Wet Chemistry	Auto-analyzer Lachat Quick Chem				
164	Wet Chemistry	pH/ISE meter Orion	710A			
165	Wet Chemistry	Expandable Ion Analyzer Orion Research	EA920			
167	Wet Chemistry	Spectrophotometer (UV-VIS) Shimadzu	1201			
171	Wet Chemistry	Polarograph EG&G Princeton Applied Research	384B			
176	Wet Chemistry	Field Meter				
177	Wet Chemistry	Mettler AE200 Analytical Balance				
178	Wet Chemistry	Turbidimeter Hach	2100N			
186	Wet Chemistry	Koehler Rapid Tester Flashpoint Tester	RT-1			
187	Wet Chemistry	Mettler DL12 Auto-Titrator				
188	Wet Chemistry	YSI Conductivity Meter	3200			
189	Wet Chemistry	Lachat	FIA-8000			
194	Wet Chemistry	Total Organic Halogen: ThermoGlass	1200			
196	Wet Chemistry	Lachat IC	8000			
198	Wet Chemistry	Total Organic Carbon Analyzer, OI Analytical	1010			
205	Wet Chemistry	OHAUS TP4KD Toploading Balance				
206	Wet Chemistry	Mettler BB600 Toploading Balance				
207	Wet Chemistry	Denver Instrument A-250 Analytical Balance				
208	Wet Chemistry	Mettler AE163 Analytical Balance				





## Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
210	Wet Chemistry	Mettler AE163 Analytical Balance				
298	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model Aqua 20			
299	Wet Chemistry	OIC Available Cyanide Analyzer				
303	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model 20	1/2006	24618583	Refurb
305	Wet Chemistry	Orion 3-Star Benchtop DO Meter	Model 1113000	2/2006	7383	New
306	Wet Chemistry	Dionex Ion Chromatograph	Model ICS-2000	3/2006	6020239	New
309	Wet Chemistry	pH Meter, Fisher Accumet Basic	Model AB15	3/28/2007	AB92325491	New
310	Wet Chemistry	Market Forge Autoclave	Model STM-E	3/30/2007	226071	New
313	Wet Chemistry	HACH Turbidimeter	Model 2100N	8/1/2008	07060C022389	New
314	Wet Chemistry	VWR Forced Air Oven	Model 1370FM	10/3/2007	4104307	New
315	Wet Chemistry	Thermo Scientific TOX Analyzer	Model ECS 1200	10/18/2007	2003.481	New
321	Wet Chemistry	BW Technologies	Gas Alert Micro		Propoerty of Kent Couty DPW	
322	Wet Chemistry	Chemetrics VVR	Photometer		5121	
324	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	2/2008	E750730372E	New
326	Wet Chemistry	OI Analytical Automated Chemistry Analyzer	Flow Solution 3100 (322689/323898)	9/2008	821831887/826833549	New
198T	Wet Chemistry					
305a	Wet Chemistry	Orion DO Probe	Model 081010MD	2/2006	Lot Number RJS16	New
312a	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
312b	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
324T	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	6/17/2008		Loaner
101	Metals	ICP Spectrophotometer PE	Optima 3000			
106	Metals	Atomic Absorption Spectrophotometer Furnace	3			
114	Metals	ICP Mass Spectrometer	ELAN 6000			
116	Metals	Perkin Elmer Optima Trace ICP	3300 DV			
201	Metals	ICP Mass Spectrometer	ELAN 6100			
202	Metals	PSA Low-Level Mercury Analyzer	Millenium System			
203	Metals	A&D FX-2000 Toploading Balance				
211	Metals	Mettler PB1502 Toploading Balance				
216	Metals	PSA Cold Vapor AA Mercury Analyzer	Millenium System			
217	Metals	Env. Express Hotblock, CS154		2000	424CEC0564	
218	Metals	Env. Express Hotblock, CS154		2000	944CEC1008	
219	Metals	Env. Express Hotblock, CS154		2002	1423CEC1147	
220	Metals	Env. Express Hotblock, CS154		2002	1423CEC1113	
311	Metals	Perkin Elmer Optima ICP-OES	Model 5300DV	5/7/2007	077C7032601	New





## Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
316	Metals	Mettler Analytical Balance	Model XS204	12/21/2007	1128261601	New
144	Semivolatiles GC	Gas Chromatograph (Dual ECD)	HP-5890A			
151	Semivolatiles GC	Liquid Chromatograph Perkin Elmer	PDA 235/240 HPLC			
157	Semivolatiles GC	Gas Chromatograph HP	5890A (FID)			
158	Semivolatiles GC	Gas Chromatograph HP	5890A (ECD)			
159	Semivolatiles GC	Gas Chromatograph Varian	3400 (FID)-SV			
174	Semivolatiles GC	Gas Chromatograph HP	5890 (ECD/FID)			
199	Semivolatiles GC	Gas Chromatograph HP-6890 Dual ECD				
221	Semivolatiles GC	Perkin-Elmer 200 LC Plus HPLC				
222	Semivolatiles GC	Agilent 6890 Dual ECD				
325	Semivolatiles GC	Clarus Gas Chromatograph	Model 500	7/2008	5564	New
133	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
138	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
195	Semivolatiles MS	HP 5973 Quadrupole Mass Spectrometer				
304	Semivolatiles MS	MS = Agilent MSD	Model 5975B	9/7/2006	MS = US60522528 GC = HP 6890N SN US10626080	New
308	Semivolatiles MS	MS = Agilent MSD	Model 5975B	2/2007	US65125179 GC = HP 6890N SN CN10703067	New
117	Volatiles GC	Agilent PID/ELCD GC	6890			
140	Volatiles GC	HP GC	5890 Series II			
142	Volatiles GC	HP GC	5890 Series II			
132	Volatiles MS	GC/MS Varian Ion Trap	Saturn II			
139	Volatiles MS	HP Quadrupole GC/MS	5971			
145	Volatiles MS	HP Quadrupole GC/MS	GC-5890/MS-5971			
197	Volatiles MS	HP 5973 Quadrupole GC/MS				
204	Volatiles MS	Mettler BB2440 Toploading Balance				
224	Volatiles MS	Agilent 5973 Inert MSD				
302	Volatiles MS	Branson Heated Ultrasonic Cleaner	3210R-MTH			
323	Volatiles MS	Agilent GC/MS	Model 6890/5973 Inert	2/21/2008	CN10426060/US35120404	New



# Appendix D





## New Employee Orientation Checklist

Name: \_\_\_\_\_ Employee #: \_\_\_\_\_ Date of Hire: \_\_\_\_/\_\_\_\_/\_\_\_\_

### ***I. Personnel Information Review (Human Resources Manager)***

Reviewed (☑)	Item
	Employee Information Sheet Completed
	I-9 Employment Eligibility Verification Form Completed
	W-4 Forms Completed
	Employee Benefits Reviewed
	Direct Deposit Forms Initiated
	Details of Compensation Reviewed
	Key Fob to the Facility Provided (Number _____ )
	Employee Handbook Distributed
	Code of Ethics / Data Integrity Policy Agreement Form Signed and Collected. Violation of Ethics Policy Explained.

Signatures below attest that all the information or items described above have been discussed/provided:

\_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Human Resources Manager Signature

\_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Employee Signature





## New Employee Orientation Checklist

### *II. Quality Assurance Training (Quality Assurance Officer)*

Reviewed (☑)	Item
	Initial and Continuing Demonstration of Capability Requirements Reviewed
	Corrective Action (Non-Conformance) Investigation Procedure Reviewed
	Error Correction Policy Reviewed
	Code of Ethics/Data Integrity Policies Explained
	Initials Added to the Initials Logbook
	Training Forms Initiated for the Following Documents:  QA Manual  Corrective Action SOP, GR-10-106 or GR-03-101 or GR-03-124  Manual Integration SOP, GR-10-115  General Guidelines for Data Validation and Reporting, GR-10-103  Internal Chain-of-Custody, GR-10-104  Data Confidentiality, GR-10-118

Signatures below attest that all the information or items described above have been discussed/provided:

\_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Quality Assurance Officer Signature

\_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Employee Signature





## New Employee Orientation Checklist

### *III. Safety Training (Health and Safety Officer)*

Reviewed (☑)	Item
	MSDS Location Discussed
	Safety Walk/Safety Equipment Review, First-Aid Cabinet Locations Identified
	Safety Exam Explained-First two of thirteen videos completed (others to be completed on own during normal working hours)
	Training Forms Initiated for the Following Documents:  Chemical Hygiene Plan  Safety Manual Copy  Emergency Action Plan Copy
	Safety Glasses Ordered or Distributed

Signatures below attest that all the information or items described above have been discussed/provided:

\_\_\_\_/\_\_\_\_/\_\_\_\_ \_\_\_\_\_/\_\_\_\_/\_\_\_\_



# Appendix E





## CODE OF ETHICS / DATA INTEGRITY AGREEMENT

All full time, part time and contracted employees working for TriMatrix Laboratories, Inc. are required to make every effort to conduct quality work with data integrity, ethical practices and professionalism. To ensure strength in the individual, in the laboratory organization and in client relationships, each employee must be aware of the following company policies:

- I. Each TriMatrix employee is responsible for the propriety and consequences of his or her actions when representing the laboratory through sample analysis, data review, adherence to policies and procedures, client /vender relationships, other employees and/or visitors.
- II. All aspects of company business must be conducted in an ethical, legal and professional manner, and in compliance with all applicable federal, state and local laws and regulations.
- III. Under no circumstances must client confidentiality be compromised or any information regarding the client be revealed to another agency without the client's prior written permission.
- IV. Gratuities, gifts and/or rewards provided by clients or vendors are laboratory property and may not be kept for personal use without written approval.
- V. Reporting of data integrity issues is encouraged. Reporting shall be kept confidential when anonymity is requested and/or required.

Additionally, violations of the data integrity/code of ethics policy may result in immediate termination of employment with TriMatrix Laboratories, Inc. Such violations include the following:

- A. Intentionally misrepresenting laboratory data in any manner.
- B. Intentionally misapplying any date and/or time.
- C. Intentional representation of another employee without written approval.
- D. Intentional omission of any information, fact or datum.
- E. Intentional deviation from or shortcut through a procedure without written approval.

A highly ethical approach to laboratory analysis/reporting is a key component of the TriMatrix laboratory objective. This approach is backed by management in providing the facilities, equipment and time necessary minimize undue pressures to make compromises, whether such pressures be internal or external.

### AGREEMENT STATEMENT

I have read and understood the Code of Ethics/Data Integrity Agreement, and agree to abide by all policies stated. I understand that violation of these policies may result in severe consequences up to and including termination of my employment with TriMatrix Laboratories, Inc.

---

Employee (print name)

---

Signature

---

Date



# Appendix F





## New Instrument Accuracy Study

**Instrument Number:** \_\_\_\_\_ **Method Reference:** \_\_\_\_\_ **Analyst:** \_\_\_\_\_ **Date Analyzed:** \_\_\_\_\_

[illegible]





## New Instrument Accuracy Study

**Instrument Number:** \_\_\_\_\_

**Method Reference:** \_\_\_\_\_

**Analyst:** \_\_\_\_\_ **Date Analyzed:** \_\_\_\_\_

**Date Analyzed:** \_\_\_\_\_

[illegible]



# Appendix G





## New Instrument Information and Initial Demonstration of Capability

Item: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Manufacturer: \_\_\_\_\_

Date Received: \_\_\_\_\_

Model: \_\_\_\_\_

Location: \_\_\_\_\_

Initial Demonstration of Capability Passed: Yes / No / NA

Date Initial Demonstration of Capability Completed: \_\_\_\_\_

Initial Demonstration of Capability Data Attached: Yes / No / NA

Adequate Sensitivity Achieved (LFB or MDL Completed): Yes / No / NA

LFB or MDL Documentation Attached: Yes / No / NA

Date LFB or MDL Completed: \_\_\_\_\_

Linear Range Developed and Demonstrated Yes / No / NA

Linear Range Development Information Attached: Yes / No / NA

Notes: \_\_\_\_\_

### Approvals and Assigned Instrument Number

\_\_\_\_\_  
Quality Assurance Manager

\_\_\_\_\_  
Laboratory Area Manager

TriMatrix Instrument Number: \_\_\_\_\_

Date In Service: \_\_\_\_\_



# Appendix H





## DEMONSTRATION OF CAPABILITY FOR \*\*\*\*\*

[illegible]



# Appendix I





# INORGANIC LABORATORY DEMONSTRATION OF CAPABILITY

Parameter: Percent Solids

Trainer: John Doe

Method: SW-846 3550B/GR-07-115

Trainee: John Smith

Analyst	Date	Run #1	Run #2	Run #3	Run #4	Units	Inst. #	Standard Deviation	Average	Degrees of Freedom D	Experimental Student's t Value	Tabular Student's t Value	Are the Two Sets of Results Statistically the Same AND RSDs<20?
John Doe	12/31/02	48.3	55.6	44.2	47.5	%	117	4.81	48.9	5.48	0.227	4.032	YES(PASS)
John Smith	12/31/02	45.9	50.2	52.1	44.7	%	117	3.50	48.2				



# Appendix J





## NELAC Demonstration of Capability Certification Statement

Employee Name: \_\_\_\_\_ Date: \_\_\_\_\_

Method Name(s), Number(s), and Revision(s):  
\_\_\_\_\_

Matrix: \_\_\_\_\_ Analyte(s) or Parameter(s): \_\_\_\_\_

SOP Number: \_\_\_\_\_ Revision Number: \_\_\_\_\_

**We, the undersigned, CERTIFY that:**

Yes / NA

☐ ☐

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

☐ ☐

2. The test method(s) was performed by the analyst identified on this certification.

☐ ☐

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

☐ ☐

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

With *true* meaning consistent with supporting data; *accurate* meaning based on good laboratory practices consistent with sound scientific principles/practices; *complete* meaning includes the results of all supporting performance testing; and *self explanatory* meaning data properly labeled and stored so that the results are clear and require no additional explanation.

☐ ☐

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

**This certification form must be completed each time an Initial Demonstration of Capability study is performed, or when a Continuing Demonstration of Capability study is performed in conjunction with a revised SOP.**

\_\_\_\_\_  
Area Supervisor  
Heather L. Brady

\_\_\_\_\_  
Date

\_\_\_\_\_  
Quality Assurance Department  
Tom C. Boocher

\_\_\_\_\_  
Date



# Appendix K



## LABORATORY TRAINING CHECKLIST

Employee Name: \_\_\_\_\_  
 Instructor Name: \_\_\_\_\_  
 Method Number(s) and  
 Revision(s): \_\_\_\_\_  
 SOP Name, Number, and  
 Revision: \_\_\_\_\_  
 Applicable Matrices: \_\_\_\_\_

n/a	Trainer/Trainee Initials		CheckPoint Item
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	1) The employee has read the method and the standard operating procedure.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	2) The instructor has reviewed the method and the procedure with the employee.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	3) The instructor has performed a manual demonstration of the procedure.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	4) The employee has correctly performed the procedure under direct supervision.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	5) The employee has correctly performed the procedure without direct supervision.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	6) The employee has successfully and exclusively completed an Initial Demonstration of Capability (IDC).
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	7) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
<input checked="" type="checkbox"/>	<input type="text"/>	<input type="text"/>	8) If applicable, or a MDL study does not yet exist, the employee has successfully completed a MDL study for all applicable matrixes.
<input checked="" type="checkbox"/>	<input type="text"/>	<input type="text"/>	9) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	10) The employee has been instructed in the QA/QC requirements of this procedure.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	11) The employee has been instructed in the proper procedure governing paperflow, benchsheet completion, and other relevant documentation requirements.
<input type="checkbox"/>	<input type="text"/>	<input type="text"/>	12) NELAC Demonstration of Capability Certification Statement is Attached.

*The required CheckPoints have been successfully completed, and in my opinion this employee has been adequately trained to correctly perform this procedure.*

**Instructor:** \_\_\_\_\_ **Date:** \_\_\_\_\_

*I have read and understand the SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.*

**Employee:** \_\_\_\_\_ **Date:** \_\_\_\_\_



# Appendix L





### METHOD DETECTION LIMIT STUDY

[illegible]









Laboratories, Inc.

**INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY**

2008 WATER/SOIL

## METHOD DETECTION LIMIT VERIFICATION STUDY

[illegible]



# Appendix M





## SOP MAJOR REVISION LABORATORY TRAINING CHECKLIST

**Employee Name:** \_\_\_\_\_  
**Method Number(s) and** \_\_\_\_\_  
**SOP Name, Number, and** \_\_\_\_\_  
**Revision:** \_\_\_\_\_  
**Applicable Matrices:** \_\_\_\_\_

n/a	Employee Initials	CheckPoint Item
<input type="checkbox"/>	<input type="text"/>	1) I have read the updated method and/or the revised Standard Operating Procedure.
<input type="checkbox"/>	<input type="text"/>	2) I have successfully completed an Initial Demonstration of Capability (IDC).
<input type="checkbox"/>	<input type="text"/>	3) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
<input type="checkbox"/>	<input type="text"/>	4) If applicable, or a MDL study does not yet exist, I have successfully completed a MDL study for all applicable matrixes.
<input type="checkbox"/>	<input type="text"/>	5) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
<input type="checkbox"/>	<input type="text"/>	6) I have been instructed in any new QA/QC requirements of this procedure.
<input type="checkbox"/>	<input type="text"/>	7) NELAC Demonstration of Capability Certification Statement is Attached.

*The required CheckPoints have been successfully completed.*

**Date:** \_\_\_\_\_ **Quality Assurance:** \_\_\_\_\_

*I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.*

**Date:** \_\_\_\_\_ **Employee Signature:** \_\_\_\_\_





***SOP MINOR REVISION  
LABORATORY TRAINING CHECKLIST***

**Employee Name:** \_\_\_\_\_  
**Method Number(s) and  
Revision(s):** \_\_\_\_\_

**SOP Name, Number, and  
Revision:** \_\_\_\_\_  
**Applicable Matrices:** \_\_\_\_\_

n/a	Employee Initials	CheckPoint Item
<input type="checkbox"/>	<input type="text"/>	1) I have read and understood the updated method and/or the revised Standard Operating Procedure.
<input type="checkbox"/>	<input type="text"/>	2) I have read and understood any new QA/QC requirements of this procedure.
<input type="checkbox"/>	<input type="text"/>	3) NELAC Demonstration of Capability Certification Statement is Attached.

*I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.*

**Date:** \_\_\_\_\_ **Employee Signature:** \_\_\_\_\_

*The SOP revision has been successfully implemented.*

**Date:** \_\_\_\_\_ **QA/QC Signature:** \_\_\_\_\_



# Appendix N



# Container Packing List

 For any questions regarding these containers,  
 contact a Project Chemist at (616) 975-4500.

Client:

Project:

Page 1 of 1

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																											
			0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	
1																														
2																														
3																														
4																														
5																														
6																														
7																														
8																														
9																														
10																														
11																														
12																														
13																														
14																														
15																														
16																														
17																														
18																														
19																														
20																														
Total Containers																														

This container type requires field-filtering

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General Short Hold	Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	Plastic	125, 250, 500, 1000	pH <2 w/ H <sub>2</sub> SO <sub>4</sub>	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO <sub>3</sub>	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000NM	pH <2 w/ H <sub>2</sub> SO <sub>4</sub>	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> ; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass w/ Septa Lid	250	pH <2 w/ H <sub>2</sub> SO <sub>4</sub>	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H <sub>2</sub> SO <sub>4</sub>	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H <sub>2</sub> SO <sub>4</sub>	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO <sub>3</sub>	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10 mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WWs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe
	26	Drinking Water Volatiles	40 mL Clear Glass Vial	40	Ascorbic Acid at Lab; HCl in Field	Yellow

Notes:

		DI Water for Equipment Blanks	Qty.
		Container Type and Size	
		VOC Free	
		Millipore	
		ASTM Metals Free	





Project Chemist Initials	Added to Calendar & Folders (initials/date)	Revision:	Revised By/Date:
--------------------------	---	-----------	------------------

Client: \_\_\_\_\_ Project Manager: \_\_\_\_\_  
Project: \_\_\_\_\_ Contact: \_\_\_\_\_  
TriMatrix Project No: \_\_\_\_\_ Date of Request: \_\_\_\_\_

Type of Order: ☐ One-Time  $\Rightarrow$  Due to Client: \_\_\_\_\_ ☐ AM ☒ PM  
or

☒ Calendar  $\Rightarrow$  Frequency: ☐ Weekly ☐ Semi-Annually  
☐ Monthly ☐ Annually  
☐ Quarterly ☒ Daily

Prepare Containers For:

Months	<input type="checkbox"/> Jan	<input type="checkbox"/> Feb	<input type="checkbox"/> Mar	<input type="checkbox"/> Apr	<input type="checkbox"/> May	<input type="checkbox"/> Jun
	<input type="checkbox"/> Jul	<input type="checkbox"/> Aug	<input type="checkbox"/> Sep	<input type="checkbox"/> Oct	<input type="checkbox"/> Nov	<input type="checkbox"/> Dec
Weeks	<input type="checkbox"/> 1	<input type="checkbox"/> 2	<input type="checkbox"/> 3	<input type="checkbox"/> 4	<input type="checkbox"/> 5	
Days	<input type="checkbox"/> M	<input type="checkbox"/> T	<input type="checkbox"/> W	<input type="checkbox"/> TH	<input type="checkbox"/> F	

Containers will be ☒ Picked Up or ☐ Shipped via: ☐ First Overnight ☒ Standard Overnight  
☐ Priority Overnight ☐ Express Saver  
☐ 2-Day ☐ Ground  
☐ Saturday Delivery ☐ TriMatrix Courier  
☐ Other: \_\_\_\_\_

**Pick up/Ship Date:** \_\_\_\_\_

Ship Containers to: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Telephone No: \_\_\_\_\_

☐ Shipment to be billed to FedEx Account No.:

Shipment to include: ☐ COCs (Qty) \_\_\_\_\_ ☐ Custody Seals ☐ Temperature Blanks  
☐ MSDS Sheets for all preservatives used ☐ WB TM#? ☐ Y ☒ N  
Comments: ☐ Cooler Banding Required  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Assembled by/Date:		Checked by/Date:	Shipped by/Date:
Cooler Number(s) Used:	Coolers Sealed With Tape      Banding Strap	Tracking Number Label(s):	
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		
	<input type="checkbox"/> <input type="checkbox"/>		

TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, Grand Rapids, MI 49512 (616)975-4500



# Appendix O



# Sample Receipt Record



**TriMatrix**  
Laboratories, Inc.

**Date:** \_\_\_\_\_

Delivery Method **A**: \_\_\_\_\_ No. of Sample Boxes: \_\_\_\_\_ Number of Coolers: \_\_\_\_\_ Signed for By: \_\_\_\_\_ Time: \_\_\_\_\_

Delivery Method **B**: \_\_\_\_\_ No. of Sample Boxes: \_\_\_\_\_ Number of Coolers: \_\_\_\_\_ Signed for By: \_\_\_\_\_ Time: \_\_\_\_\_

Delivery Method **C**: \_\_\_\_\_ No. of Sample Boxes: \_\_\_\_\_ Number of Coolers: \_\_\_\_\_ Signed for By: \_\_\_\_\_ Time: \_\_\_\_\_

Delivery Method **D**: \_\_\_\_\_ No. of Sample Boxes: \_\_\_\_\_ Number of Coolers: \_\_\_\_\_ Signed for By: \_\_\_\_\_ Time: \_\_\_\_\_

TriMatrix Courier (TC): \_\_\_\_\_ No. of Sample Boxes: \_\_\_\_\_ Number of Coolers: \_\_\_\_\_ Signed for By: \_\_\_\_\_ Time: \_\_\_\_\_

Page/ Line Number	Client	Quantity of Coolers OR TriMatrix Cooler Number	Arrived in Laboratory					Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM	Received By	Delivery Method Letter		
1-1									
1-2									
1-3									
1-4									
1-5									
1-6									
1-7									
1-8									
1-9									
1-10									
1-11									
1-12									
1-13									
1-14									
1-15									
1-16									
1-17									
1-18									
1-19									
1-20									
1-21									



# Appendix P



# SAMPLE RECEIVING / LOG-IN CHECKLIST

Client	Project-Submittal No.	
Receipt Record Page/Line No.	new / add to	Project Chemist
	Sample Nos.	

## Coolers Received

Recorded by (initials/date)	<input type="checkbox"/> Cooler <input type="checkbox"/> Box <input type="checkbox"/> Other _____	Qty Received	<input type="checkbox"/> IR Gun (#202) Thermometer Used <input type="checkbox"/> Digital Thermometer (#54) <input type="checkbox"/> See Additional Cooler Information Form <input type="checkbox"/> Other (# _____)
-----------------------------	---	--------------	---

Cooler No.	Time	Cooler No.	Time	Cooler No.	Time	Cooler No.	Time
Custody Seals <input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact		Custody Seals <input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact		Custody Seals <input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact		Custody Seals <input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact	
Coolant Location: Dispersed / Top / Middle / Bottom		Coolant Location: Dispersed / Top / Middle / Bottom		Coolant Location: Dispersed / Top / Middle / Bottom		Coolant Location: Dispersed / Top / Middle / Bottom	
Coolant/Temperature Taken Via: <input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers		Coolant / Temperature Taken Via: <input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers		Coolant / Temperature Taken Via: <input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers		Coolant / Temperature Taken Via: <input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers	
Alternate Temperature Taken Via: <input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container		Alternate Temperature Taken Via: <input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container		Alternate Temperature Taken Via: <input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container		Alternate Temperature Taken Via: <input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container	
Recorded °C	Correction Factor °C	Actual °C	Recorded °C	Correction Factor °C	Actual °C	Recorded °C	Correction Factor °C
tb			tb			tb	
tb location: representative / in ice		tb location: representative / in ice		tb location: representative / in ice		tb location: representative / in ice	
1			1			1	
2			2			2	
3			3			3	
Average °C		Average °C		Average °C		Average °C	
<input type="checkbox"/> Cooler ID on COC?		<input type="checkbox"/> Cooler ID on COC?		<input type="checkbox"/> Cooler ID on COC?		<input type="checkbox"/> Cooler ID on COC?	
<input type="checkbox"/> VOC trip blank received?		<input type="checkbox"/> VOC trip blank received?		<input type="checkbox"/> VOC trip blank received?		<input type="checkbox"/> VOC trip blank received?	

**If any shaded areas checked, complete Sample Receiving Non-Conformance Form**

## Paperwork Received

N/A	Yes	No	
	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> No COC received
	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> Chain of Custody Record(s)?
	<input type="checkbox"/>	<input type="checkbox"/>	If No, COC initiated by _____
	<input type="checkbox"/>	<input type="checkbox"/>	Rec'd for Lab signed/date/time?
	<input type="checkbox"/>	<input type="checkbox"/>	Shipping Document?
	<input type="checkbox"/>	<input type="checkbox"/>	Other _____

COC ID Nos.

☐ TriMatrix

☐ Other (name or ID#)

## Check COC for Accuracy

Yes	No	
<input type="checkbox"/>	<input checked="" type="checkbox"/>	Sample ID matches COC?
<input type="checkbox"/>	<input checked="" type="checkbox"/>	Sample date and time matches COC?
<input type="checkbox"/>	<input type="checkbox"/>	Container type completed on COC?
<input type="checkbox"/>	<input checked="" type="checkbox"/>	All container types indicated are received?

## Sample Condition Summary

N/A	Yes	No	
	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Broken containers/lids?
	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Missing or incomplete labels?
	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Illegible information on labels?
	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Low volume received?
	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Inappropriate containers received?
	<input type="checkbox"/>	<input type="checkbox"/>	VOC vials / TOX containers have headspace?
	<input type="checkbox"/>	<input type="checkbox"/>	Extra sample locations / containers not listed on COC?

## Check Sample Preservation

N/A	Yes	No	
	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Average sample temperature ≤ 6° C?
	<input type="checkbox"/>	<input type="checkbox"/>	Completed Sample Preservation Verification Form?
	<input type="checkbox"/>	<input checked="" type="checkbox"/>	Samples preserved correctly?
	<input type="checkbox"/>	<input type="checkbox"/>	If "No", added orange tag?
	<input type="checkbox"/>	<input type="checkbox"/>	Received pre-preserved VOC soils?
		<input type="checkbox"/> MeOH <input type="checkbox"/> Na <sub>2</sub> SO <sub>4</sub>	

## Check for Short Hold-Time Prep/Analyses

<input type="checkbox"/> Bacteriological
<input type="checkbox"/> Air Bags
<input type="checkbox"/> EnCores / Methanol Pre-Preserved
<input type="checkbox"/> Formaldehyde/Aldehyde
<input type="checkbox"/> Green-tagged Containers
<input type="checkbox"/> Yellow/White-tagged 1L Ambers (SV Prep-Lab)

### AFTER HOURS ONLY:

COPIES OF COC TO LAB AREA(S)

☐ NONE RECEIVED

☐ RECEIVED, COCs TO LAB(S)

## Notes

<input type="checkbox"/> Trip blank received	<input type="checkbox"/> Trip blank not listed on COC
<input type="checkbox"/> No COC received, Proj. Chemist reviewed (init./date) _____	
<input type="checkbox"/> No analysis requested, Proj. Chemist completed (init./date) _____	

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤ 1 Hour Goal Met?
		Yes / No



Project Chemist Use		Log-In Use	
<b>Notify Laboratory Personnel of Short Hold-Times and/or Rush Work</b> <span style="float: right;"><input type="checkbox"/> NONE</span> (Lab personnel notified/date) <input type="checkbox"/> Inorganics _____ <input type="checkbox"/> Microbiology (bacteria) _____ <input type="checkbox"/> Metals Prep _____ <input type="checkbox"/> Metals _____ <input type="checkbox"/> GC-Volatiles _____ <input type="checkbox"/> MS-Volatiles _____ <input type="checkbox"/> Semi-Vol. Prep _____ <input type="checkbox"/> GC-Semi-Volatiles _____ <input type="checkbox"/> MS-Semi-Volatiles _____		<b>Log Samples into LIMS</b> <span style="float: right; border: 1px solid black; padding: 2px;">Sample Nos.</span> <div style="display: flex; justify-content: space-between;"> <span>N/A</span> <span>Yes</span> </div> <input type="checkbox"/> Receive samples in LIMS <input type="checkbox"/> Date/Time received entered in LIMS match COC <input type="checkbox"/> Read project and submittal narratives <input type="checkbox"/> Enter VOC rack/tray number into submittal narrative <input type="checkbox"/> Enter sample information into LIMS <input type="checkbox"/> Add any sample narratives <input type="checkbox"/> If non-conformance issues, add sample qualifiers <input type="checkbox"/> Print sample number labels	
<b>Log-In Priority</b> <span style="margin-left: 20px;"><input type="checkbox"/> RUSH</span> <span style="margin-left: 20px;"><input type="checkbox"/> Standard</span>		<b>Log-in Analyst (initials/date/time)</b>	
<b>Project Chemist Notes to Log-In Personnel</b> Trip Blank: <span style="margin-left: 20px;"><input type="checkbox"/> Log-in</span> <span style="margin-left: 20px;"><input type="checkbox"/> Do not log-in</span> <input type="checkbox"/> Prep Storage Blank for Client (VOCs) <input type="checkbox"/> Sub-Contracting required <span style="margin-left: 20px;"><input type="checkbox"/> Coolant required</span> <input type="checkbox"/> Non-TriMatrix or non-standard container type(s) received Check pH of container type _____ Expected pH: _____ <span style="margin-left: 20px;"><input type="checkbox"/> Adjust if needed</span> <input type="checkbox"/> Adjust pH of orange-tagged containers <input type="checkbox"/> Lab-filter samples and document on Preservation Form		<b>Label Sample containers</b> <div style="display: flex; justify-content: space-between;"> <span>N/A</span> <span>Yes</span> <span>No</span> </div> <input type="checkbox"/> <input type="checkbox"/> LIMS label matches tag? <input type="checkbox"/> <input type="checkbox"/> <b>DISCREPANCIES CORRECTED IN LIMS</b> Initials/Date: _____ <input type="checkbox"/> <input type="checkbox"/> Applicable stickers applied to labels? <div style="margin-left: 40px;"> <input type="checkbox"/> MS/MSD sample  <input type="checkbox"/> Composite before analysis           </div> <input type="checkbox"/> <input type="checkbox"/> Applicable stickers applied to containers? <div style="margin-left: 40px;"> <input type="checkbox"/> Waste sample  <input type="checkbox"/> PT sample  <input type="checkbox"/> USDA regulated           </div> <input type="checkbox"/> <input type="checkbox"/> Orange-tagged containers present? <input type="checkbox"/> <input type="checkbox"/> Adjust pH per Project Chemist <input type="checkbox"/> <input type="checkbox"/> Initials and Date/Time Adjusted on orange tag? <input type="checkbox"/> <input type="checkbox"/> Initials and Date/Time Adjusted on Preservation Form?	
		<b>Verify Label Accuracy</b> <input type="checkbox"/> Second analyst checked labels for accuracy? <input type="checkbox"/> <input type="checkbox"/> Verify that Orange-tagged containers adjusted/initialialed?	
<b>Labeled by (initials/date)</b>		<b>Verified by (initials/date)</b>	
<b>Sample Narratives to be added at Log-in</b> <div style="border-bottom: 1px solid black; height: 20px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 20px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 20px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 20px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 20px; margin-bottom: 5px;"></div>		<b>Sample Storage</b> <span style="float: right;">Check all that apply</span> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;">           bacteria.....            non-volatiles.....            volatiles.....            waste.....            waste VOCs.....            low-level Hg ....         </div> <div style="width: 45%;"> <input type="checkbox"/> bacteria refrigerator  <input type="checkbox"/> walk-in cooler  <input type="checkbox"/> volatile lab refrigerator  <input type="checkbox"/> waste cabinet  <input type="checkbox"/> log-in hood refrigerator  <input type="checkbox"/> metals lab - DO NOT STORE IN WALK-IN         </div> </div>	
		<b>Paperwork</b> <div style="display: flex; justify-content: space-between;"> <span>N/A</span> <span>Yes</span> </div> <input type="checkbox"/> original COC (white) <input type="checkbox"/> copy of COC (yellow) <input type="checkbox"/> receiving/log-in checklist <input type="checkbox"/> additional cooler information form <input type="checkbox"/> sample preservation verification <input type="checkbox"/> sample receiving non-conformance form <input type="checkbox"/> shipping documents <input type="checkbox"/> custody seals <input type="checkbox"/> arrival log <input type="checkbox"/> other (note)	



# Appendix Q



# SAMPLE RECEIVING / LOG-IN CHECKLIST

## ADDITIONAL COOLER INFORMATION

Recorded by (initials/date)			Client			Project-Submittal No.		
			Receipt Log No.			Sample Nos.		

Cooler No.		Time	
Custody Seals			
<input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact			
Coolant Location:			
Dispersed / Top / Middle / Bottom			
Coolant/Temperature Taken Via:			
<input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers			
Alternate Temperature Taken Via:			
<input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container			
Recorded °C	Correction Factor °C	Actual °C	
tb			
tb location: representative / in ice			
1			
2			
3			
Average °C			
<input type="checkbox"/> Cooler ID on COC? <input type="checkbox"/> VOC trip blank received?			

Cooler No.		Time	
Custody Seals			
<input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact			
Coolant Location:			
Dispersed / Top / Middle / Bottom			
Coolant / Temperature Taken Via:			
<input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers			
Alternate Temperature Taken Via:			
<input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container			
Recorded °C	Correction Factor °C	Actual °C	
tb			
tb location: representative / in ice			
1			
2			
3			
Average °C			
<input type="checkbox"/> Cooler ID on COC? <input type="checkbox"/> VOC trip blank received?			

Cooler No.		Time	
Custody Seals			
<input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact			
Coolant Location:			
Dispersed / Top / Middle / Bottom			
Coolant / Temperature Taken Via:			
<input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers			
Alternate Temperature Taken Via:			
<input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container			
Recorded °C	Correction Factor °C	Actual °C	
tb			
tb location: representative / in ice			
1			
2			
3			
Average °C			
<input type="checkbox"/> Cooler ID on COC? <input type="checkbox"/> VOC trip blank received?			

Cooler No.		Time	
Custody Seals			
<input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact			
Coolant Location:			
Dispersed / Top / Middle / Bottom			
Coolant/Temperature Taken Via:			
<input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers			
Alternate Temperature Taken Via:			
<input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container			
Recorded °C	Correction Factor °C	Actual °C	
tb			
tb location: representative / in ice			
1			
2			
3			
Average °C			
<input type="checkbox"/> Cooler ID on COC? <input type="checkbox"/> VOC trip blank received?			

Cooler No.		Time	
Custody Seals			
<input type="checkbox"/> none <input type="checkbox"/> present / intact <input type="checkbox"/> present / not intact			
Coolant Location:			
Dispersed / Top / Middle / Bottom			
Coolant/Temperature Taken Via:			
<input type="checkbox"/> loose ice / avg 2-3 containers <input type="checkbox"/> bagged ice / avg 2-3 containers <input type="checkbox"/> blue ice / avg 2-3 containers <input checked="" type="checkbox"/> none / avg 2-3 containers			
Alternate Temperature Taken Via:			
<input type="checkbox"/> temperature blank (tb) <input type="checkbox"/> 1 container			
Recorded °C	Correction Factor °C	Actual °C	
tb			
tb location: representative / in ice			
1			
2			
3			
Average °C			
<input type="checkbox"/> Cooler ID on COC? <input type="checkbox"/> VOC trip blank received?			

Comments



# Appendix R



Client		Project-Submittal No.	
Receipt Log No.	Completed By (initials/date)	Project Chemist	

COC ID No.				Adjusted by: _____				DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
				Date: _____							
Container Type	5	4	13		3	6	15				
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe				
Preservative	NaOH	H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>		None	HNO <sub>3</sub>	HNO <sub>3</sub>				
Expected pH	>12	<2	<2		~7	<2	<2				
COC Line No. 1											
COC Line No. 2											
COC Line No. 3											
COC Line No. 4											
COC Line No. 5											
COC Line No. 6											
COC Line No. 7											
COC Line No. 8											
COC Line No. 9											
COC Line No. 10											

pH strip lot No.  
☐ **HC896537**  
☐ \_\_\_\_\_

**Aqueous Samples:** For each sample and container type, check the box if pH is acceptable. **If pH is not acceptable for any sample container, record pH in box, and note on Sample Receiving Checklist and on Sample Receiving Non-Conformance Form.** If approved by Project Chemist, add acid or base to the sample to achieve the correct pH. Add up to, but do not exceed 2x the volume initially added at container prep (see table below for initial volumes used). **Add orange pH tag to sample container and record information requested. Record adjusted pH on this form. Do not adjust pH for container types 3, 6, and 15.**

COC ID No.				Adjusted by: _____				DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
				Date: _____							
Container Type	5	4	13		3	6	15				
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe				
Preservative	NaOH	H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>		None	HNO <sub>3</sub>	HNO <sub>3</sub>				
Expected pH	>12	<2	<2		~7	<2	<2				
COC Line No. 1											
COC Line No. 2											
COC Line No. 3											
COC Line No. 4											
COC Line No. 5											
COC Line No. 6											
COC Line No. 7											
COC Line No. 8											
COC Line No. 9											
COC Line No. 10											

Container Size (mL)	Original Vol. of Preservative (mL)
<hr/>	
Container Type 5: NaOH	
500	2.5
1000	5.0
<hr/>	
Container Type 4: H <sub>2</sub> SO <sub>4</sub>	
125	0.5
250	1.0
500	2.0
1000	4.0
<hr/>	
Container Type 13: H <sub>2</sub> SO <sub>4</sub>	
500	2.5



# Appendix S





# SAMPLE RECEIVING NON-CONFORMANCE REPORT

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

List non-conformance issues associated with this submittal in the chart below/left. Identify discrepancies between the COC and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.

Type of Problem													and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.										
COC ID No.	Line No.	Discrepancy	Missing Container	Broken Container	Label Missing / Incomplete	Label Illegible	Low Volume	Inappropriate Container	Headspace	Not Listed on COC	Preservation	COC					Sample Tag					Line Item Comments	
												Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty		

General Comments:	Project Chemist (initials/date)
-------------------	---------------------------------



# Appendix T



Client:	Project Manager: <b>Rick D. Wilburn</b>
Project: <b>TCLP Semi-Volatiles</b>	Project Number: <b>[none]</b>
Work Order: <b>TCLP October 2008</b>	SDG:

**Report To:**

R

T

2

L

Phone:

Fax:

**Invoice To:**

TriMatrix Laboratories

Mr. Rick D. Wilburn

5560 Corporate Exchange Court SE

Grand Rapids, MI 49512-5503

Phone: 616-975-4500 x4

Fax: 616-942-7463

Client Due Date: **Nov-24-08 23:00 (21 day TAT)**

Date Received: Oct-24-08 12:00

Date Logged In: Oct-27-08 08:15

W.O. Comments: *QC is 3MD.*Report Level: **3MD**

Received By: Rick D. Wilburn

Logged In By: William D. Cole

Analysis	Lab Due Date	TAT	Expires	Analysis Comments
----------	--------------	-----	---------	-------------------

**0810557-01 TCLP Semi-Volatiles [Soil]***Sampled Oct-23-08 08:00 Eastern by*

8270C TCLP Herbs	Nov-24-08 17:00	10	Oct-30-08 08:00
8270C TCLP SVOC/Pest	Nov-24-08 17:00	10	Oct-30-08 08:00
TCLP Organics Extraction	Nov-24-08 17:00	10	Nov-06-08 08:00

**0810557-02 TCLP Analytes in Soil [Soil]***Sampled Oct-23-08 08:00 Eastern by*

8151A Herbicides CLP [dual-col]	Nov-24-08 17:00	10	Nov-06-08 08:00
8270C Standard SVOCs	Nov-24-08 17:00	10	Nov-06-08 08:00
Solids, Total 3550B (%)	Nov-24-08 17:00	10	Nov-06-08 08:00



Client:  
 Project: **TCLP Semi-Volatiles**  
 Work Order: **TCLP October 2008**

Project Manager: **Rick D. Wilburn**  
 Project Number: **[none]**  
 SDG:

## Inorganic - Wet Chemistry Analysis Detail

<u>Matrix</u>	<u>Analysis</u>	<u>Unit</u>	<u>MDL</u>	<u>RL</u>
Soil	Solids, Total 3550B (%)	%	0.1	0.1

\* indicates custom

## Semivolatiles GC Analysis Detail

<u>Analyte</u>	<u>CLrept?</u>	<u>QCrept?</u>	<u>MDL</u>	<u>RL</u>
<b>Soil</b>	<b>8151A Herbicides CLP [dual-col]</b>	<b>mg/kg</b>		
2,4-D	Y	Y	0.0602	0.2
2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
2,4-D [2C]	Y	Y	0.0602	0.2
2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05

\* indicates custom

## Semivolatiles MS Analysis Detail

<u>Analyte</u>	<u>CLrept?</u>	<u>QCrept?</u>	<u>MDL</u>	<u>RL</u>
<b>Soil</b>	<b>TCLP Organics Extraction</b>			
<b>Soil</b>	<b>8270C Standard SVOCs</b>	<b>mg/kg</b>		
1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
Hexachlorobenzene	Y	Y	0.00337	0.0167
Hexachlorobutadiene	Y	Y	0.00107	0.0167
Hexachloroethane	Y	Y	0.000778	0.0167
3+4-Methylphenol	Y	Y	0.00129	0.0167
2-Methylphenol	Y	Y	0.00223	0.0167
Nitrobenzene	Y	Y	0.00199	0.0167
Pentachlorophenol	Y	Y	0.00368	0.0167
Pyridine	Y	Y	0.00566	0.0167
2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
<b>Soil</b>	<b>8270C TCLP Herbs</b>	<b>mg/L</b>		
2,4-D	Y	Y	0.00409	0.1
2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
<b>Soil</b>	<b>8270C TCLP SVOC/Pest</b>	<b>mg/L</b>		
1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
2,4-Dinitrotoluene	Y	Y	0.000214	0.005
Hexachlorobenzene	Y	Y	0.0000117	0.005
Hexachlorobutadiene	Y	Y	0.000125	0.005
Hexachloroethane	Y	Y	0.0000378	0.005
Nitrobenzene	Y	Y	0.0000257	0.005
Pyridine	Y	Y	0.000385	0.05
Pentachlorophenol	Y	Y	0.000187	0.005
2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
2,4,5-Trichlorophenol	Y	Y	0.000109	0.005

\* indicates custom



Client:  
Project: **TCLP Semi-Volatiles**  
Work Order: **TCLP October 2008**

Project Manager: **Rick D. Wilburn**  
Project Number: **[none]**  
SDG:

## Semivolatiles MS Analysis Detail

<u>Analyte</u>	<u>CLrept?</u>	<u>QCrept?</u>	* indicates custom	
			<u>MDL</u>	<u>RL</u>
2-Methylphenol	Y	Y	0.000144	0.005
3-Methylphenol	Y	Y	0.0000157	0.005
4-Methylphenol	Y	Y	0.0000157	0.005
gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
Endrin	Y	Y	0.000284	0.005
Methoxychlor	Y	Y	0.0000723	0.005
Technical Chlordane	Y	Y	0.000124	0.005
Heptachlor	Y	Y	0.0000907	0.005
Heptachlor Epoxide	Y	Y	0.0000758	0.005
Toxaphene	Y	Y	0.000293	0.5



# Appendix U



## Semivolatiles GC Sample Receipt Notice

Client:	C	Project Manager:	Rick D. Wilburn
Project:	TCLP Semi-Volatiles	Project Number:	[none]
Client Due Date:	Nov-24-08 23:00 (21 day TAT)	Report Level:	3MD
W.O. Comments:	QC is 3MD.		

Lab Number	Sample Name Analysis	Matrix	Sampled Date TAT    Expire Date		Sample Comments Lab Due Date    Comments	
------------	-------------------------	--------	------------------------------------	--	---	--

0810557-02	TCLP Analytes in Soil	Soil	Oct-23-08 08:00 Eastern			
	8151A Herbicides CLP [dual-col]	10	Nov-06-08 08:00	Nov-24-08 17:00		



**Semivolatiles GC Analysis Detail**

				* indicates custom	
<u>Analyte</u>		<u>CLrept?</u>	<u>QCrept?</u>	<u>MDL</u>	<u>RL</u>
<b>Soil</b>	<b>8151A Herbicides CLP [dual-col]</b>	<b>mg/kg</b>			
	2,4-D	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
	2,4-D [2C]	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05



## Semivolatiles MS Sample Receipt Notice

Client:	<b>T</b>	Project Manager:	<b>Rick D. Wilburn</b>
Project:	<b>TCLP Semi-Volatiles</b>	Project Number:	<b>[none]</b>
Client Due Date:	<b>Nov-24-08 23:00 (21 day TAT)</b>	Report Level:	<b>3MD</b>
W.O. Comments:	<i>QC is 3MD.</i>		

Lab Number	Sample Name Analysis	Matrix	Sampled Date		Sample Comments	
			TAT	Expire Date	Lab Due Date	Comments
<b>0810557-01</b>	<b>TCLP Semi-Volatiles</b>	<b>Soil</b>	<b>Oct-23-08 08:00 Eastern</b>			
	8270C TCLP Herbs	10	Oct-30-08 08:00	Nov-24-08 17:00		
	8270C TCLP SVOC/Pest	10	Oct-30-08 08:00	Nov-24-08 17:00		
	TCLP Organics Extraction	10	Nov-06-08 08:00	Nov-24-08 17:00		
<b>0810557-02</b>	<b>TCLP Analytes in Soil</b>	<b>Soil</b>	<b>Oct-23-08 08:00 Eastern</b>			
	8270C Standard SVOCs	10	Nov-06-08 08:00	Nov-24-08 17:00		



## Semivolatiles MS Analysis Detail

Analyte		CLrept?	QCrept?	* indicates custom	
	MDL			RL	
Soil	TCLP Organics Extraction				
Soil	8270C Standard SVOCs	mg/kg			
	1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
	2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
	Hexachlorobenzene	Y	Y	0.00337	0.0167
	Hexachlorobutadiene	Y	Y	0.00107	0.0167
	Hexachloroethane	Y	Y	0.000778	0.0167
	3+4-Methylphenol	Y	Y	0.00129	0.0167
	2-Methylphenol	Y	Y	0.00223	0.0167
	Nitrobenzene	Y	Y	0.00199	0.0167
	Pentachlorophenol	Y	Y	0.00368	0.0167
	Pyridine	Y	Y	0.00566	0.0167
	2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
	2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
Soil	8270C TCLP Herbs	mg/L			
	2,4-D	Y	Y	0.00409	0.1
	2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
Soil	8270C TCLP SVOC/Pest	mg/L			
	1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
	2,4-Dinitrotoluene	Y	Y	0.000214	0.005
	Hexachlorobenzene	Y	Y	0.0000117	0.005
	Hexachlorobutadiene	Y	Y	0.000125	0.005
	Hexachloroethane	Y	Y	0.0000378	0.005
	Nitrobenzene	Y	Y	0.0000257	0.005
	Pyridine	Y	Y	0.000385	0.05
	Pentachlorophenol	Y	Y	0.000187	0.005
	2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
	2,4,5-Trichlorophenol	Y	Y	0.000109	0.005
	2-Methylphenol	Y	Y	0.000144	0.005
	3-Methylphenol	Y	Y	0.0000157	0.005
	4-Methylphenol	Y	Y	0.0000157	0.005
	gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
	Endrin	Y	Y	0.000284	0.005
	Methoxychlor	Y	Y	0.0000723	0.005
	Technical Chlordane	Y	Y	0.000124	0.005
	Heptachlor	Y	Y	0.0000907	0.005
	Heptachlor Epoxide	Y	Y	0.0000758	0.005
	Toxaphene	Y	Y	0.000293	0.5



## Semivolatiles MS, Soil, 3550B Sonication Extraction

Surrogate #1 = 8110251 (Pre-Prep)

Batch Comments: (none)

<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>	<u>Work Order</u>	<u>Analysis</u>
0810557	8270C Standard SVOCs	0810557	8270C MDEQ BNA	0810557	8270C MDEQ Base/Neutrals
0810648	8270C MDEQ BNA	0810665	8270C MDEQ Base/Neutrals	0811070	8270C Standard SVOCs
0811070	8270C MDEQ BNA	0811070	8270C MDEQ Base/Neutrals	0811154	8270C MDEQ BNA

<i>Lab Number</i>	<i>Contain</i>	<i>Prepared</i>	<i>By</i>	<i>Initial (g)</i>	<i>Final (mL)</i>	<i>uL Surrogate</i>	<i>Source ID</i>	<i>Spike ID</i>	<i>uL Spike</i>	<i>Client / QC Type</i>	<i>Extraction Comments</i>
0813151-BLK1		Nov-10-08 07:09	BJH	30	1	100				BLANK	
0813151-BLK2		Nov-10-08 07:09	JLB	30	1	100				BLANK	mdeq base/neutrals
0813151-BLK3		Nov-10-08 07:09		30	1	100				BLANK	
0813151-BLK4		Nov-10-08 07:09		30	1	100				BLANK	
0813151-DUP1		Nov-10-08 07:09	BJH	30	1	100	0810557-02			DUPLICATE	
0813151-BS1		Nov-10-08 07:09	BJH	30	1	100		8110206	100	LCS	
0813151-BS2		Nov-10-08 07:09	BJH	30	1	100		8100124	100	LCS	
0813151-BS3		Nov-10-08 07:09	JLB	30	1	100		8110206	100	LCS	mdeq base/neutrals
0813151-BS4		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-BS5		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-MS1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE	
0813151-MSD1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE DUP	
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810648-19	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-20	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-21	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-24	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810648-25	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810665-01	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only
0810665-02	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only

Comments:	Analyst Initials:
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Semivolatiles MS, Soil, Nov-12-08

Instrument = 308, Calibration = 8K05005

## Sequence Analyses:

8270C MDEQ BNA

Lab Number	Analysis	Contain	STD ID	ISTD ID	Client / QC Type	Extraction Comments
8111315-TUN1	QC		8100195	8060452	MS TUNE	
8111315-CCV1	QC		8110293	8060452	CALIBRATION CHECK	
8111315-CCV2	QC		8110085	8060452	CALIBRATION CHECK	
0813151-BLK4	QC			8060452	BLANK	
0813151-BS5	QC			8060452	LCS	
0811154-04	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-05	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-06	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-07	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-09	8270C MDEQ BNA	A 02		8060452	Engineering	

Comments:

Analyst  
Initials:



## Lab PM (Rick D. Wilburn) Nov-11-08 - Dec-09-08

*ized,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr*

Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLF	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	



# Appendix V



# TriMatrix Laboratories, Inc. - Department

Work Orders Received Sep-01-08 to Sep-30-08 - Printed Dec-08-08 10:26 by TCB

Department	Samples	Analyses	Price	Surcharge	Total
Inorganic - Wet Chemistry	1622	5878	\$120,471.90	\$333.75	\$120,805.60
Metals	997	10085	\$83,255.61	\$829.00	\$84,084.61
Semivolatiles GC	495	638	\$60,893.00	\$110.00	\$61,003.00
Semivolatiles MS	468	548	\$78,247.00	\$58.15	\$78,305.15
Volatiles GC	116	117	\$5,438.50	\$14.40	\$5,452.90
Volatiles MS	1382	1400	\$135,669.00	\$297.75	\$135,966.80
<b>**TOTALS**</b>	<b>5080</b>	<b>18666</b>	<b>\$483,975.01</b>	<b>\$1,643.05</b>	<b>\$485,618.06</b>



# TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:36 by TCB

Department: [All]

Analysis: [All]

Matrix: [All]

Department	On-Time	Total	%
Inorganic - Wet Chemistry	4117	4399	93.6
Metals	6889	6907	99.7
Semivolatiles GC	644	658	97.9
Semivolatiles MS	390	422	92.4
Volatiles GC	147	229	64.2
Volatiles MS	1415	1438	98.4



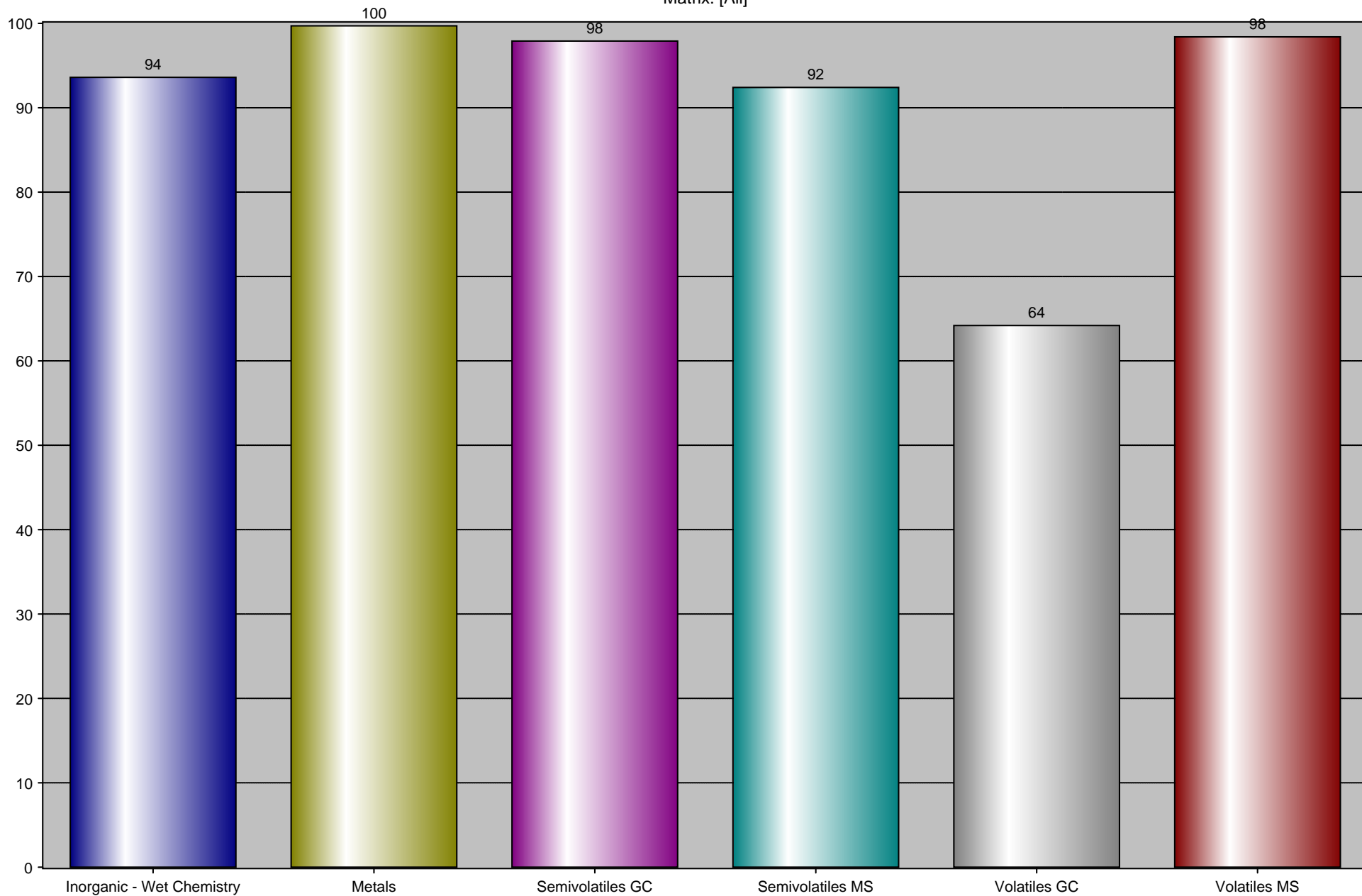
TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:34 by TCB

Department: [All]

Analysis: [All]

Matrix: [All]





**Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08***Available, Cancelled, Completed, Invoiced, Preliminary, Received, Reported*

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0707274	64/64	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Summer (35005)	RDW	22	Jul-17-07	Aug-16-07
0801455	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	19	Jan-25-08	Feb-21-08
0801456	120/120	3FL		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	21	Jan-25-08	Feb-25-08
0801501	136/136	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jan-30-08	Feb-25-08
0802130	60/60	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Winter (35005)	RDW	16	Feb-08-08	Mar-03-08
0802188	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Feb-12-08	Feb-26-08
0803447	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Mar-27-08	Apr-10-08
0804113	116/116	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring (35508)	RDW	10	Apr-07-08	Apr-21-08
0805464	1/1	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring Re-Do (35508)	RDW	5	May-21-08	May-29-08
0805489	70/70	3MD		Completed	Analytical Products Group	DMRQA Testing (36330)	RDW	26	May-21-08	Jun-27-08
0806220	4/4	3MD		Completed	Analytical Products Group	WP Performance Testing Program Quick Turn (35508)	RDW	10	Jun-11-08	Jun-25-08
0806250	75/75	3MD		Completed	TriMatrix Laboratories	pH Strip Testing (36236)	RDW	10	Jun-12-08	Jun-26-08
0807484	126/126	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jul-23-08	Aug-18-08
0807485	120/120	3MD		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	22	Jul-24-08	Aug-25-08
0807486	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	17	Jul-24-08	Aug-18-08
0808052	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-04-08	Aug-18-08
0808059	2/2	3MD		Completed	Analytical Products Group	DMRQA Testing Micro (36330)	RDW	10	Aug-05-08	Aug-19-08
0808244	48/48	2RLM		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-13-08	Aug-27-08
0808571	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-28-08	Sep-12-08
0809070	10/10	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	17	Sep-04-08	Sep-29-08
0809419	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Sep-22-08	Oct-06-08
0810124	110/110	3MD		Completed	Analytical Products Group	WP Performance Testing Program Fall (35508)	RDW	18	Oct-08-08	Nov-03-08
0810557	6/6	3MD		Completed	RTC	TCLP Semi-Volatiles ([none])	RDW	21	Oct-24-08	Nov-24-08



**Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08***Available, Cancelled, Completed, Invoiced, Preliminary, Received, Reported*

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0810558	4/4	3MD		Completed	RTC	Minnesota DRO/GRO ([none])	RDW	21	Oct-24-08	Nov-24-08



**Lab PM (Rick D. Wilburn) Nov-01-08 - Nov-30-08***ized,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr*

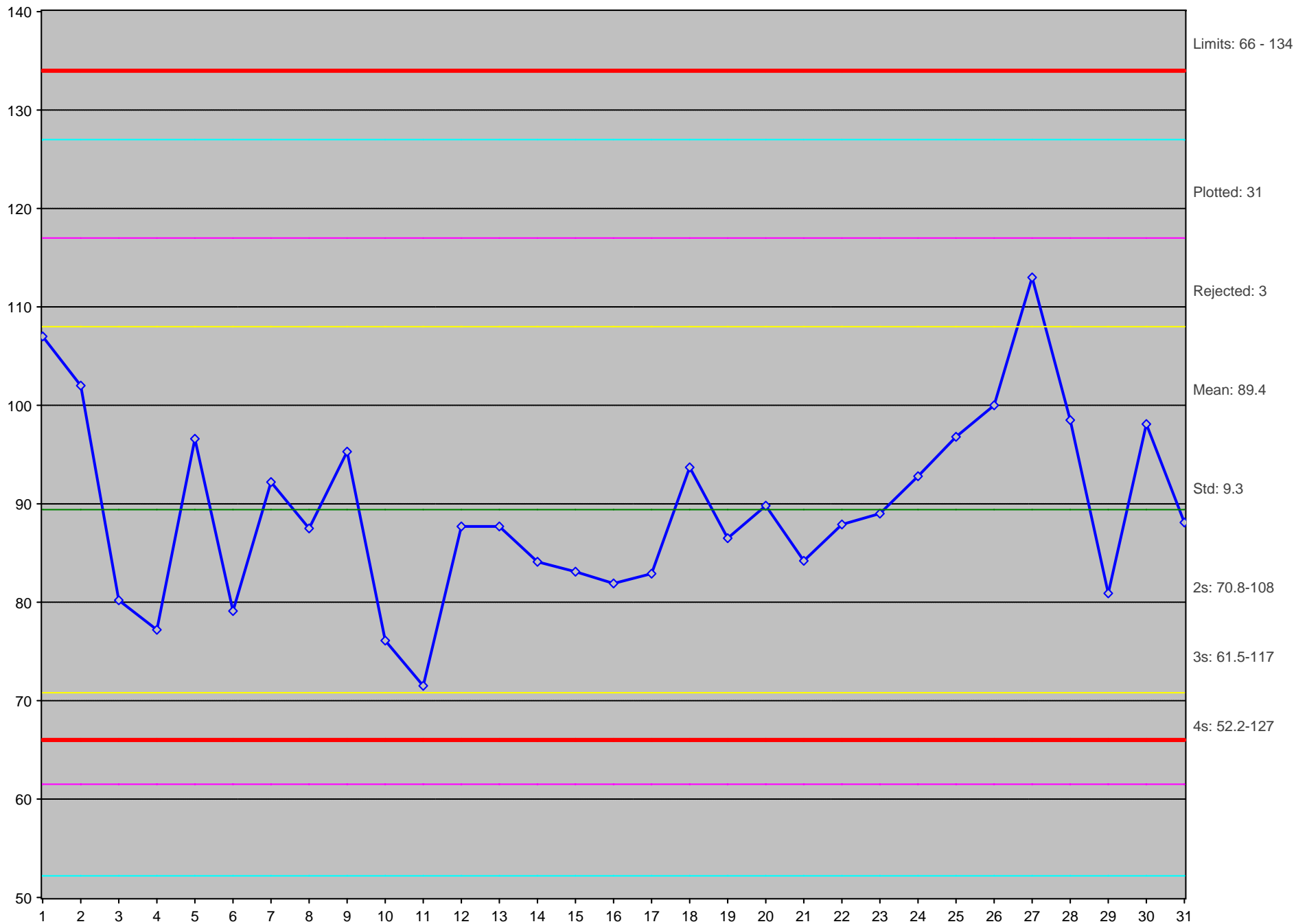
Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLF	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%)	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	



# Appendix W



TriMatrix Laboratories, Inc. - LCS %R for CHRYSENE  
8270C Standard SVOCs IN Water Printed: Dec-08-08 11:04 by TCB  
All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]





Printed: Dec-08-08 11:09

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

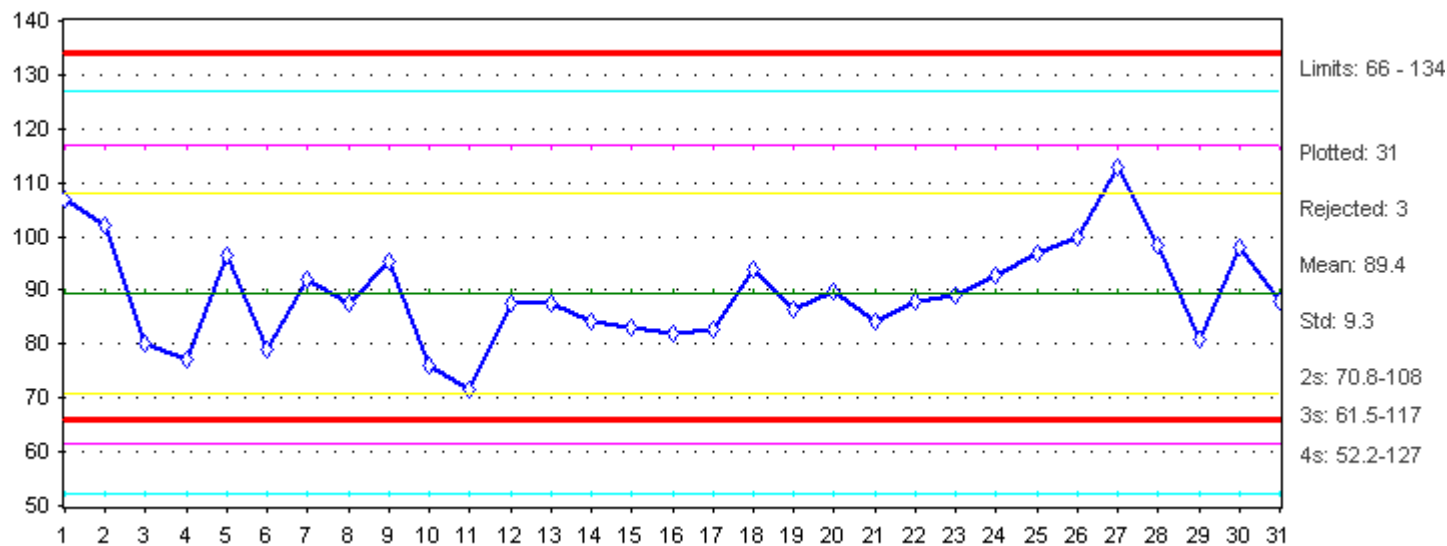
Analyses: 8270C Standard SVOCs

Analyzed By: All Analysts

Extractions: All Extractions

## Chrysene

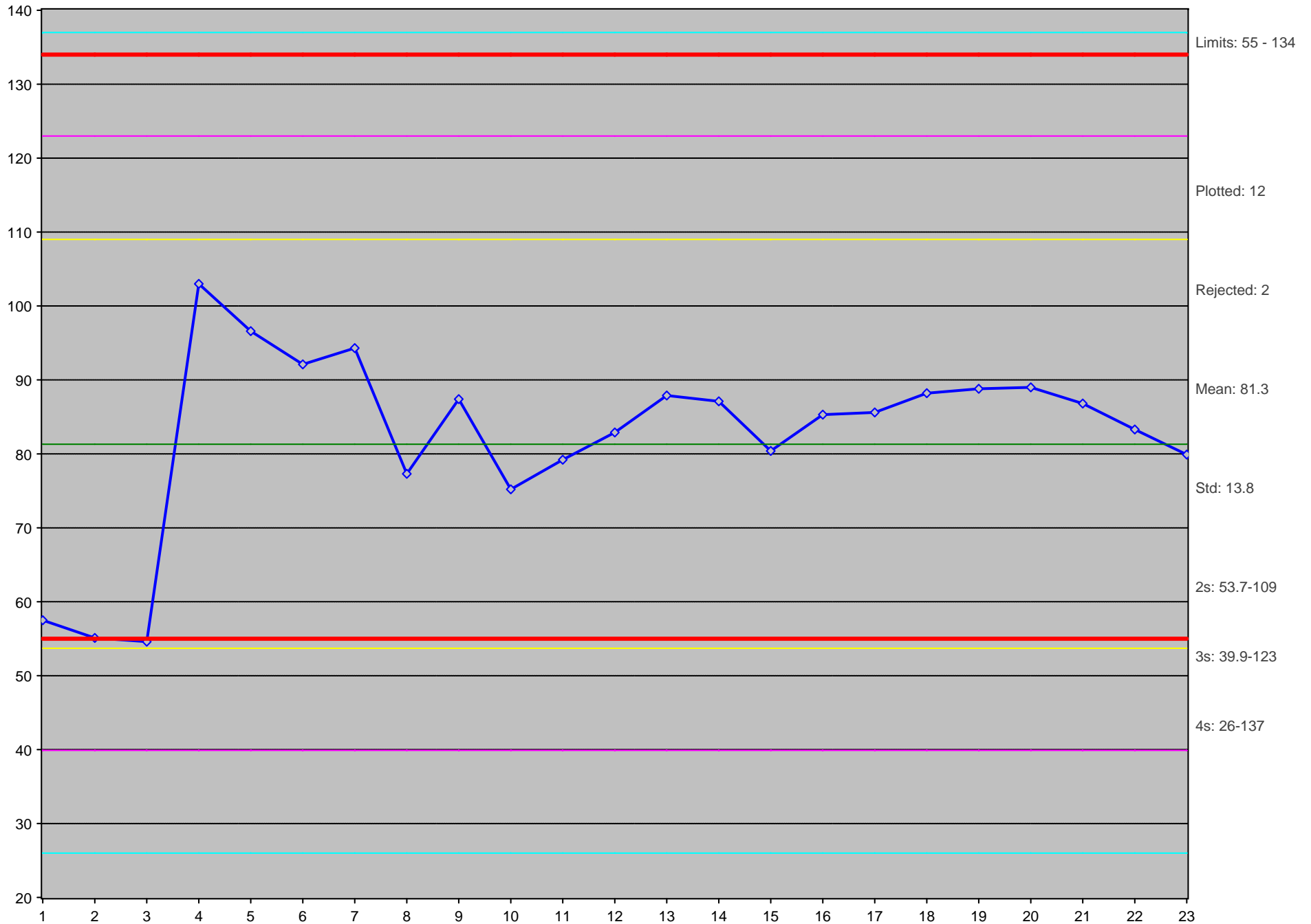
## LCS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	0809978-BS1	8/29/08	9/2/08	9.6 ug/L	10.25	106.7708	66-134	
	0810083-BS2	9/2/08	9/3/08	9.6 ug/L	9.832	102.4167	66-134	
X	0810084-BS1	9/2/08	9/4/08	9.6 ug/L	0		66-134	
	0810561-BS1	9/12/08	9/17/08	96 ug/L	76.98	80.1875	66-134	
	0810624-BS1	9/15/08	9/16/08	9.6 ug/L	7.41	77.1875	66-134	
	0810739-BS1	9/17/08	9/18/08	96 ug/L	92.76	96.625	66-134	
X	0810740-BS1	9/17/08	9/22/08	9.6 ug/L	0		66-134	
	0810938-BS1	9/22/08	9/26/08	9.6 ug/L	7.59	79.0625	66-134	
	0810561-BS2	9/22/08	9/23/08	9.6 ug/L	8.852	92.20833	66-134	GN020
	0810967-BS1	9/23/08	9/26/08	96 ug/L	83.96	87.45833	66-134	
	0811030-BS1	9/24/08	9/27/08	96 ug/L	91.5	95.3125	66-134	
	0811107-BS1	9/25/08	9/27/08	9.6 ug/L	7.31	76.14583	66-134	
X	0811107-BS2	9/25/08	10/3/08	9.6 ug/L	0		66-134	
	0811170-BS1	9/26/08	9/27/08	9.6 ug/L	6.86	71.45833	66-134	
	0811170-BS2	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811170-BS3	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811030-BS2	10/1/08	10/1/08	9.6 ug/L	8.07	84.06249	66-134	
	0811661-BS1	10/9/08	10/10/08	9.6 ug/L	7.98	83.125	66-134	
	0811711-BS1	10/13/08	10/13/08	9.6 ug/L	7.866	81.9375	66-134	
	0811661-BS2	10/14/08	10/15/08	9.6 ug/L	7.96	82.91666	66-134	
	0811858-BS2	10/15/08	10/16/08	96 ug/L	89.94	93.6875	66-134	
	0811858-BS1	10/15/08	10/20/08	9.6 ug/L	8.3	86.45833	66-134	
	0812029-BS1	10/20/08	10/25/08	9.6 ug/L	8.62	89.79166	66-134	
	0812131-BS1	10/21/08	10/21/08	9.6 ug/L	8.08	84.16666	66-134	
	0812166-BS1	10/21/08	10/22/08	9.6 ug/L	8.44	87.91666	66-134	
	0812461-BS1	10/27/08	10/30/08	9.6 ug/L	8.54	88.95833	66-134	
	0812462-BS1	10/27/08	10/29/08	9.6 ug/L	8.91	92.81249	66-134	



TriMatrix Laboratories, Inc. - MS %R for CHRYSENE  
8270C Standard SVOCs IN Water Printed: Dec-08-08 11:12 by TCB  
All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]





Matrices: Water

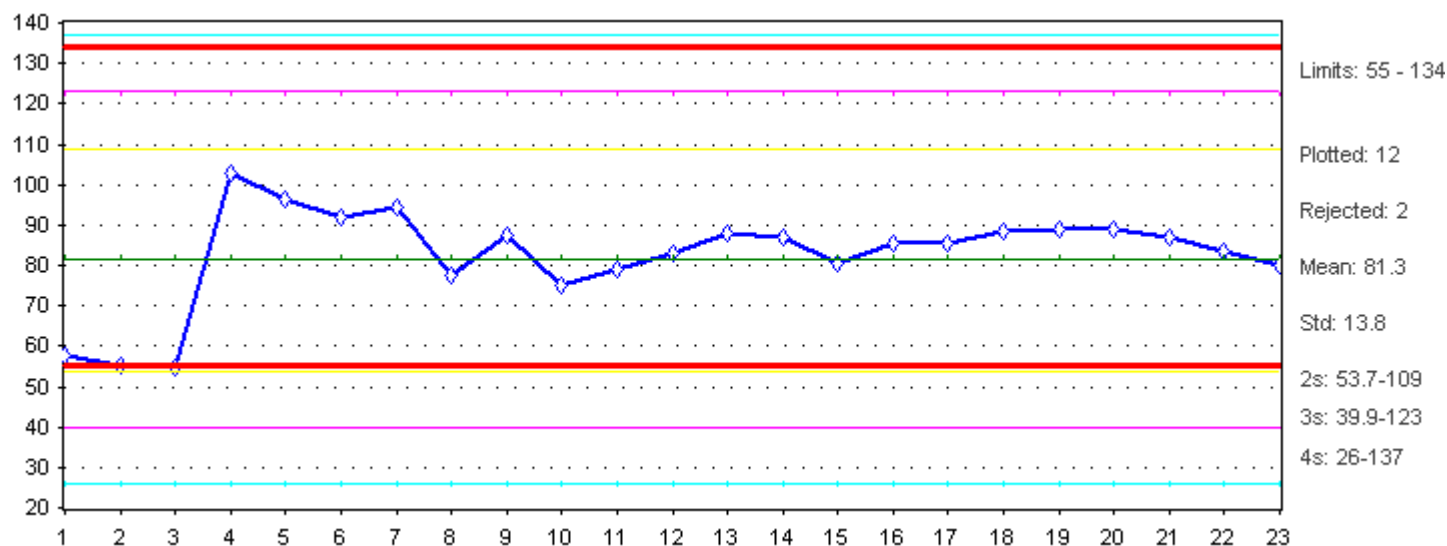
Instruments: All Instruments

Prepared By: All Extractionists

Analyzed By: All Analysts

Extractions: All Extractions

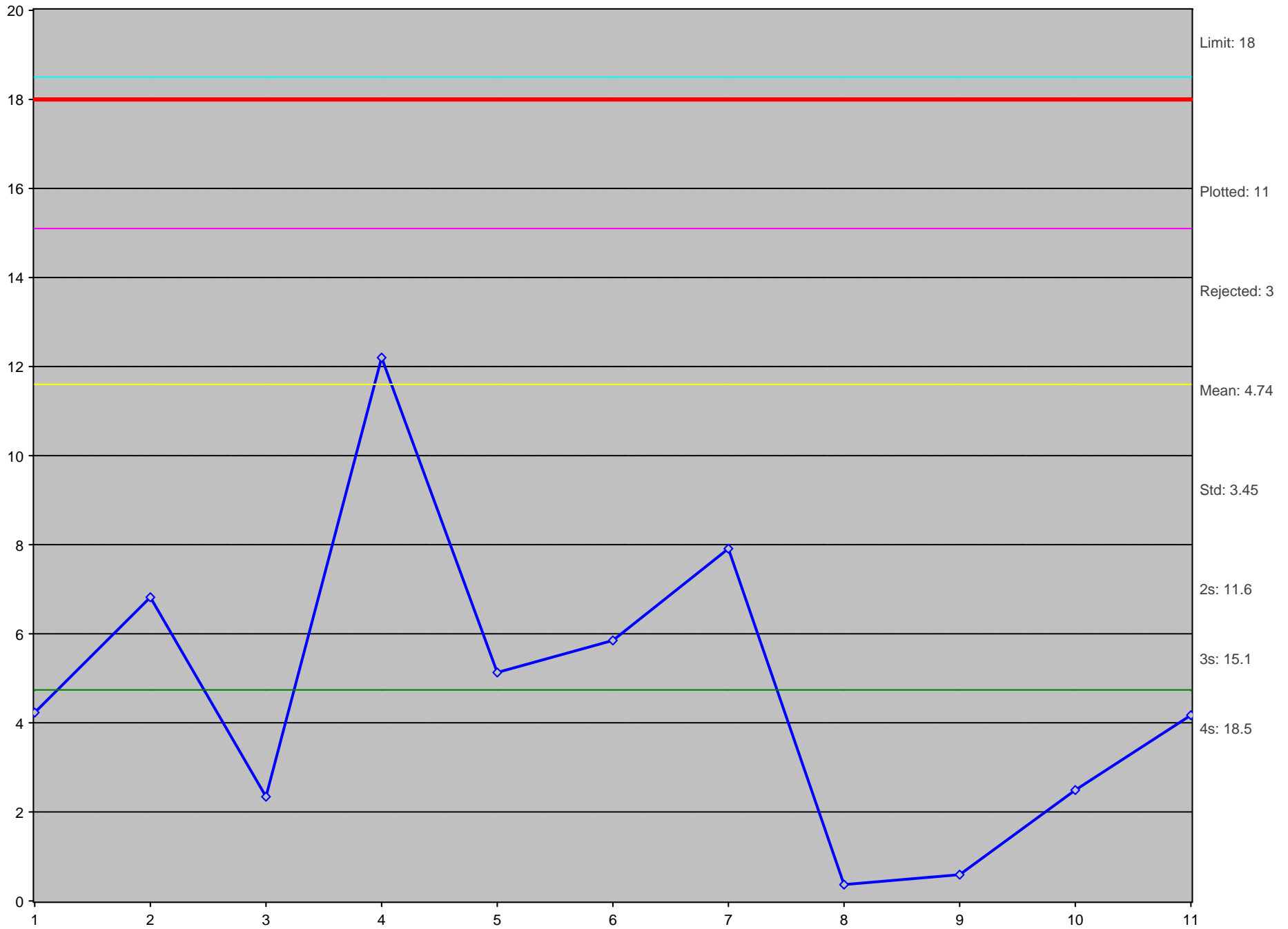
## MS %R



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Source	Limits	Qualifiers
	0809978-MS1	8/29/08	9/2/08	9.69697 ug/L	5.606061	57.5	0808570-08 (3.030303E-02)	55-134	
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.104178	0808570-08 (3.030303E-02)	55-134	
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	0.520833	0808570-29 (0.05)	55-134	
	0810084-MS1	9/2/08	9/5/08	9.6 ug/L	5.29	54.58333	0808570-29 (0.05)	55-134	
	0810561-MS1	9/12/08	9/18/08	96.9697 ug/L	100.2828	103.4166	0809136-03 (ND)	55-134	
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	0809136-03 (ND)	55-134	
	0810561-MS2	9/12/08	9/18/08	97.95918 ug/L	90.2347	92.11459	0809136-05 (ND)	55-134	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	0809136-05 (ND)	55-134	
	0810967-MS1	9/23/08	9/27/08	97.95918 ug/L	75.7551	77.33333	0809311-06 (ND)	55-134	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	0809311-06 (ND)	55-134	
	0811107-MS1	9/25/08	9/27/08	9.795918 ug/L	7.367347	75.20833	0809435-05 (ND)	55-134	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	0809435-05 (ND)	55-134	
	0811661-MS1	10/9/08	10/10/08	10.90909 ug/L	9.045455	82.91668	0810133-04 (ND)	55-134	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	0810133-04 (ND)	55-134	
	0811711-MS1	10/13/08	10/13/08	9.795918 ug/L	8.529592	87.07291	0810180-03	55-134	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	0810180-03	55-134	
	0811858-MS1	10/15/08	10/16/08	9.6 ug/L	8.19	85.31249	0810220-33 (ND)	55-134	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	0810220-33 (ND)	55-134	
	0812029-MS1	10/20/08	10/25/08	9.69697 ug/L	8.555556	88.22917	0810321-06	55-134	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	0810321-06	55-134	
	0812461-MS1	10/27/08	10/29/08	9.795918 ug/L	8.714286	88.95833	0810435-08 (ND)	55-134	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	0810435-08 (ND)	55-134	
X	0812462-MS1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0813364-MS1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
	0813840-MS1	11/24/08	12/2/08	9.494949 ug/L	7.909091	83.29787	0811481-04 (ND)	55-134	
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	0811481-04 (ND)	55-134	



TriMatrix Laboratories, Inc. - MS/MSD RPD for CHRYSENE  
8270C Standard SVOCs IN Water Printed: Dec-08-08 11:15 by TCB  
All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]





Printed: Dec-08-08 11:15

Matrices: Water

Client: All Clients

Instruments: All Instruments

Project: All Projects

Prepared By: All Extractionists

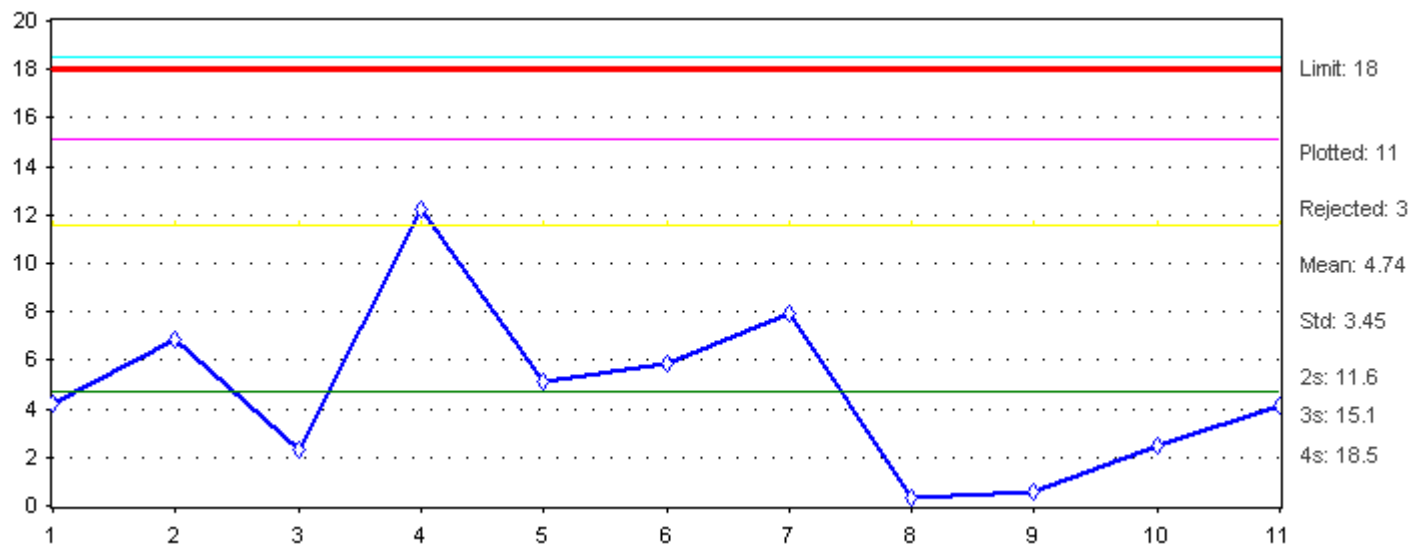
Analyses: 8270C Standard SVOCs

Analyzed By: All Analysts

Extractions: All Extractions

## Chrysene

## MS/MSD RPD



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Dup	Dup %R	Source	RPD	Limit	Qualifier
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.10417	5.606061	57.5	08570-08 (3.030303E-0.231825	18		
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	-0.5208333	5.29	54.58333	0808570-29 (0.05)	18		
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	100.2828	103.4166	0809136-03 (ND)	3.822545	18	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	90.2347	92.11459	0809136-05 (ND)	2.335835	18	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	75.7551	77.33333	0809311-06 (ND)	12.19327	18	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	7.367347	75.20833	0809435-05 (ND)	5.128205	18	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	9.045455	82.91668	0810133-04 (ND)	5.853655	18	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	8.529592	87.07291	0810180-03	7.909465	18	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	8.19	85.31249	0810220-33 (ND)	.365639	18	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	8.555556	88.22917	0810321-06	.588577	18	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	8.714286	88.95833	0810435-08 (ND)	2.489625	18	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0		0810452-01 (ND)	18		
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0		0811231-07	18		
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	7.909091	83.29787	0811481-04 (ND)	4.172097	18	



# Appendix X





## Daily Log Sheet

Thermometer Serial #: 1353

Adjustments/Observations/Comments



# Appendix Y





## Balance Calibration Verification Acceptance Window Calculations

<b>Balance ID:</b> 204	<b>Calibration Source:</b> External	
<b>Manufacturer:</b> Mettler	<b>Calibration Weight (g):</b> 1000	
<b>Serial Number:</b> J58563	<b>Calibration Weight Error (g):</b> 0.00171811	
<b>Model Number:</b> BB2440	<b>Location:</b> Volatiles Laboratory; South Bench Top	

### I. Calibration Weight Correction Calculations

Calibration Verification Weight Nominal Mass (g)	Calibration Verification Weight Correction Factor (g)	Calibration Verification Weight Actual Mass (g)	Linear Error of Balance (g)	Calibration Verification Weight Expected Mass (g)	Calibration Weight Combinations Used For Verification	
					Nominal	Actual
0.5000	0.00000190	0.5000	-0.00000086	0.5000	0.50	0.5000
5.0000	0.00000910	5.0000	-0.00000859	5.0000		
20.0000	0.00003540	20.0000	-0.00003436	20.0000	0.50 + 5 + 20	25.5000
100.0000	0.00006020	100.0001	-0.00017181	99.9999	0.50 + 100	100.4999

### II. 20 Measurements Using Each Calibration Verification Mass

Date	Replicate Number	Mass 1 (g) 0.50	Mass 2 (g) 25.50	Mass 3 (g) 100.50			
1/29/2008	1	0.49	25.48	100.49			
1/29/2008	2	0.49	25.49	100.48			
1/29/2008	3	0.48	25.47	100.48			
1/29/2008	4	0.49	25.48	100.49			
1/29/2008	5	0.48	25.48	100.48			
1/29/2008	6	0.49	25.51	100.49			
1/29/2008	7	0.52	25.49	100.49			
1/30/2008	8	0.52	25.49	100.49			
1/30/2008	9	0.50	25.48	100.48			
1/30/2008	10	0.48	25.50	100.49			
1/30/2008	11	0.50	25.48	100.48			
1/30/2008	12	0.49	25.49	100.49			
1/30/2008	13	0.48	25.50	100.50			
1/30/2008	14	0.49	25.49	100.49			
1/31/2008	15	0.51	25.50	100.50			
1/31/2008	16	0.51	25.49	100.49			
1/31/2008	17	0.48	25.52	100.51			
1/31/2008	18	0.52	25.51	100.49			
1/31/2008	19	0.50	25.52	100.48			
1/31/2008	20	0.51	25.51	100.49			

### III. Calibration Verification Acceptance Window Calculations

<b>Standard Deviation:</b>	0.01424411	0.01429022	0.00788069			
<b>Random Error:</b>	0.04273234	0.04287067	0.02364207			
<b>Acceptance Window Low:</b>	0.46	25.46	100.48			
<b>Acceptance Window High:</b>	0.54	25.54	100.52			





**TriMatrix**  
Laboratories, Inc.

## Daily Balance Calibration Logbook

<b>Balance ID:</b>	<u>204</u>	<b>Serial #:</b>	<u>J58563</u>	<b>Calibration Source:</b>	<u>External</u>	<b>Location:</b>	<u>Volatiles Laboratory;</u>
<b>Manufacturer:</b>	Mettler	<b>Model #:</b>	BB2440	<b>Calibration Weight (g):</b>	1000		South Bench Top

[illegible]



# Appendix Z



**Analytical Standard Record**  
**TriMatrix Laboratories, Inc.**  
**7120763**

Description:	8260 1 UG/L 12-28-07	Expires:	Feb-15-08
Standard Type:	Calibration Standard	Prepared:	Dec-28-07
Solvent:	WTR	Prepared By:	Diane L. VanMale
Final Volume (mls):	100	Department:	Volatiles MS
Vials:	1	Last Edit:	Dec-28-07 15:42 by DLV

Analyte	CAS Number	Concentration	Units
4-Methyl-2-pentanone (MIBK)	108-10-1	0.001	ppm
1-Chlorohexane	544-10-5	0.001	ppm
2,2-Dichloropropane	594-20-7	0.001	ppm
2-Butanone (MEK)	78-93-3	0.001	ppm
2-Chloroethyl Vinyl Ether	110-75-8	0.001	ppm
2-Chlorotoluene	95-49-8	0.001	ppm
2-Hexanone	591-78-6	0.001	ppm
2-Methylnaphthalene	91-57-6	0.001	ppm
4-Bromofluorobenzene	460-00-4	0.04	ppm
Carbon Disulfide	75-15-0	0.001	ppm
4-Isopropyltoluene	99-87-6	0.001	ppm
1,3-Dichloropropane	142-28-9	0.001	ppm
Acetone	67-64-1	0.001	ppm
Acrolein	107-02-8	0.001	ppm
Acrylonitrile	107-13-1	0.001	ppm
Benzene	71-43-2	0.001	ppm
Bromobenzene	108-86-1	0.001	ppm
Bromochloromethane	74-97-5	0.001	ppm
Bromodichloromethane	75-27-4	0.001	ppm
Bromoform	75-25-2	0.001	ppm
1,1,1,2-Tetrachloroethane	630-20-6	0.001	ppm
4-Chlorotoluene	106-43-4	0.001	ppm
1,2-Dibromo-3-chloropropane	96-12-8	0.001	ppm
1,1,1-Trichloroethane	71-55-6	0.001	ppm
1,1,2,2-Tetrachloroethane	79-34-5	0.001	ppm
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.001	ppm
1,1,2-Trichloroethane	79-00-5	0.001	ppm
1,1-Dichloroethane	75-34-3	0.001	ppm
1,1-Dichloroethene	75-35-4	0.001	ppm
1,1-Dichloropropene	563-58-6	0.001	ppm

Reviewed By	Date
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**Analytical Standard Record**  
**TriMatrix Laboratories, Inc.**  
**7120763**

1,2,3-Trichlorobenzene	87-61-6	0.001	ppm
1,2,3-Trichloropropane	96-18-4	0.001	ppm
1,4-Dichlorobenzene	106-46-7	0.001	ppm
1,2,4-Trimethylbenzene	95-63-6	0.001	ppm
1,3-Dichloropropene (Total)	542-75-6	0.002	ppm
1,2-Dibromoethane	106-93-4	0.001	ppm
1,2-Dichlorobenzene	95-50-1	0.001	ppm
1,2-Dichloroethane	107-06-2	0.001	ppm
1,2-Dichloroethane-d4	17060-07-0	0.04	ppm
1,2-Dichloroethene (Total)	540-59-0	0.002	ppm
1,2-Dichloropropane	78-87-5	0.001	ppm
1,3,5-Trimethylbenzene	108-67-8	0.001	ppm
1,3-Dichlorobenzene	541-73-1	0.001	ppm
Carbon Tetrachloride	56-23-5	0.001	ppm
1,2,4-Trichlorobenzene	120-82-1	0.001	ppm
Total Trihalomethanes		0.004	ppm
n-Butylbenzene	104-51-8	0.001	ppm
n-Propylbenzene	103-65-1	0.001	ppm
Naphthalene	91-20-3	0.001	ppm
sec-Butylbenzene	135-98-8	0.001	ppm
Styrene	100-42-5	0.001	ppm
tert-Butylbenzene	98-06-6	0.001	ppm
Tetrachloroethene	127-18-4	0.001	ppm
Tetrahydrofuran	109-99-9	0.001	ppm
Bromomethane	74-83-9	0.001	ppm
Toluene-d8	2037-26-5	0.04	ppm
Methyl tert-Butyl Ether	1634-04-4	0.001	ppm
trans-1,2-Dichloroethene	156-60-5	0.001	ppm
trans-1,3-Dichloropropene	10061-02-6	0.001	ppm
trans-1,4-Dichloro-2-butene	110-57-6	0.001	ppm
Trichloroethene	79-01-6	0.001	ppm
Trichlorofluoromethane	75-69-4	0.001	ppm
Vinyl Acetate	108-05-4	0.001	ppm
Vinyl Chloride	75-01-4	0.001	ppm
Xylene (Total)	1330-20-7	0.003	ppm
Xylene, Meta + Para	136777-61-2	0.002	ppm
Toluene	108-88-3	0.001	ppm
Dichlorofluoromethane	75-43-4	0.001	ppm
Chlorobenzene	108-90-7	0.001	ppm

Reviewed By

Date



**Analytical Standard Record**  
**TriMatrix Laboratories, Inc.**  
**7120763**

Chloroethane	75-00-3	0.001	ppm
Chloroform	67-66-3	0.001	ppm
Chloromethane	74-87-3	0.001	ppm
cis-1,2-Dichloroethene	156-59-2	0.001	ppm
cis-1,3-Dichloropropene	10061-01-5	0.001	ppm
Cyclohexane	110-82-7	0.001	ppm
Dibromochloromethane	124-48-1	0.001	ppm
Dibromofluoromethane	1868-53-7	0.04	ppm
Methylene Chloride	75-09-2	0.001	ppm
Dichlorodifluoromethane	75-71-8	0.001	ppm
Methylcyclohexane	108-87-2	0.001	ppm
Ethyl Ether	60-29-7	0.001	ppm
Ethylbenzene	100-41-4	0.001	ppm
Heptane	142-82-5	0.001	ppm
Hexachlorobutadiene	87-68-3	0.001	ppm
Hexachloroethane	67-72-1	0.001	ppm
Iodomethane	74-88-4	0.001	ppm
Isopropylbenzene	98-82-8	0.001	ppm
Methyl Acetate	79-20-9	0.001	ppm
Xylene, Ortho	95-47-6	0.001	ppm
Dibromomethane	74-95-3	0.001	ppm

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7090571	8260 Centurian Working Surrogate	Sep-13-07	Diane L. VanMale	Aug-31-08	Sep-18-07 10:47 by DLV	0.1
7120760	8260 Working Standard B	12-28-07	Diane L. VanMale	Feb-15-08	Dec-28-07 15:20 by DLV	0.001

Reviewed By

Date



Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	VO7. -1													
2	VO7. -2													
3	VO7. -3													
4	VO7. -4													
5	VO7. -5													
6	VO7. -6													
7	VO7. -7													
8	VO7. -8													
9	VO7. -9													
10	VO7. -10													
11	VO7. -11													
12	VO7. -12													
13	VO7. -13													
14	VO7. -14													
15	VO7. -15													
16	VO7. -16													
17	VO7. -17													
18	VO7. -18													



# Appendix AA





## Pipet Calibration Verification Acceptance Window Calculations

**Pipet ID:** SPK-15  
**Manufacturer:** Socorex  
**Model Number:** Calibra 822.1000  
**Serial Number:** 10111410

**Balance Used:** IN-1  
**Manufacturer:** Mettler  
**Model Number:** AE-163  
**Serial Number:** B86211

### I. 20 Weight (g) Measurements Using Each Pipet Calibration Mass

Date	Replicate Number	Volume 1 uL 100	Volume 2 uL 200	Volume 3 uL 250	Volume 4 uL 300	Volume 5 uL 500	Volume 6 uL 1000
05/04/01	1	0.0975	0.1978	0.2455	0.2924	0.4904	0.9819
05/04/01	2	0.0981	0.1978	0.2461	0.2944	0.5077	0.9958
05/04/01	3	0.0987	0.1980	0.2458	0.2945	0.5028	1.0019
05/04/01	4	0.0983	0.1973	0.2475	0.2933	0.5041	1.0011
05/04/01	5	0.1002	0.1986	0.2479	0.2936	0.4993	1.0030
05/04/01	6	0.0997	0.1984	0.2474	0.2945	0.5001	1.0006
05/04/01	7	0.1000	0.1973	0.2471	0.2942	0.5005	1.0034
05/07/01	8	0.0983	0.1965	0.2449	0.2940	0.4950	0.9930
05/07/01	9	0.0975	0.1971	0.2451	0.2936	0.4939	0.9922
05/07/01	10	0.0970	0.1933	0.2440	0.2948	0.4943	0.9943
05/07/01	11	0.0972	0.1970	0.2447	0.2927	0.4939	0.9938
05/07/01	12	0.0973	0.1963	0.2452	0.2935	0.4935	0.9928
05/07/01	13	0.0966	0.1970	0.2445	0.2939	0.4934	0.9935
05/07/01	14	0.0977	0.1961	0.2438	0.2937	0.4935	0.9920
05/08/01	15	0.0992	0.1969	0.2464	0.2973	0.4937	0.9884
05/08/01	16	0.0990	0.1970	0.2463	0.2953	0.4913	0.9918
05/08/01	17	0.0989	0.1959	0.2479	0.2977	0.4924	0.9841
05/08/01	18	0.0981	0.2012	0.2474	0.2963	0.4932	0.9858
05/08/01	19	0.0985	0.1954	0.2469	0.2962	0.4948	0.9856
05/08/01	20	0.0990	0.1976	0.2462	0.2975	0.4930	0.9865

### II. Pipet Calibration Acceptance Window Calculations

<b>Standard Deviation:</b>	0.00100755	0.00151653	0.00128600	0.00156309	0.00469383	0.00647504
<b>Random Error:</b>	0.00302265	0.00454960	0.00385799	0.00468928	0.01408148	0.01942513
<b>Average Percent Recovery</b>	98.3%	98.6%	98.4%	98.2%	99.2%	99.3%
<b>Acceptance Window Low:</b>	0.0970	0.1955	0.2461	0.2953	0.4859	0.9806
<b>Acceptance Window High:</b>	0.1030	0.2045	0.2539	0.3047	0.5141	1.0194





## Metals Laboratory Spiking Pipet Calibration Logbook

Pipet ID	Calibration Volume	Acceptance Window (g)	Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:	
			g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail
B-8	20 uL	0.0192-0.0208										
	50 uL	0.0495-0.0505										
	100 uL	0.0981-0.1019										
SPK-5	10 uL	0.0096-0.0104										
	25 uL	0.0245-0.0255										
	50 uL	0.0485-0.0515										
	100 uL	0.0982-0.1018										
SPK-12	4.00 mL	3.91-4.09										
	8.00 mL	7.84-8.16										
	9.00 mL	8.84-9.16										
	10.00 mL	9.85-10.15										
SPK-15	100 uL	0.0970-0.1030										
	200 uL	0.1955-0.2045										
	250 uL	0.2461-0.2539										
	300 uL	0.2953-0.3047										
	500 uL	0.4859-0.5141										
	1000 uL	0.9806-1.0194										
SPK-16	100 uL	0.0953-0.1047										
	200 uL	0.1944-0.2056										
	250 uL	0.2457-0.2543										
	300 uL	0.2918-0.3082										
	500 uL	0.4922-0.5078										
	1000 uL	0.9641-1.0359										



# Appendix AB





UNCONTROLLED COPY

## STANDARD OPERATING PROCEDURE

### Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1  
SW-846 Method 7470A

#### APPROVALS:

Area Supervisor: Denise S. Coffey Date: 9-12-08  
Denise S. Coffey

QA Officer: Tom C. Boocher Date: 9-11-08  
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 9/12/08  
Jeff P. Glaser

Procedure Number: GR-01-140  
Revision Number: 0.3

Date Initiated: 2/19/03  
Effective Date: 10/15/08

Date Revised: 8/4/08  
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed

Reviewed by

Review Expires

_____	_____	_____
_____	_____	_____
_____	_____	_____



SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
SW-846 Method 7470A, EPA Method 245.1  
SOP Number: **GR-01-140**

page 2 of 21

Revision Number: 0.3  
Date Revised: 8/4/08  
Date Initiated: 2/19/03

## 1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.
- 1.2 The minimum reporting limit is 0.2 ug/L.

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"*
- 2.2 *Methods for the Determination of Metals in Environmental Samples, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994*

## 3.0 SUMMARY OF PROCEDURE


- 3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.
- 3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.
- 3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.
- 3.4 Inorganic mercury is converted to mercury in the Hg<sup>2+</sup> state during the digestion, for detection and quantitation.


## 4.0 PARAMETER OR COMPOUND LIST

- 4.1 Mercury

## 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision
- 5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

Approved By:  9-11-08  
QA Officer

Approved By:  9-12-08  
Area Supervisor



# Appendix AC





## Sample Collection, Packing and Return

All supplied containers are pre-cleaned, no additional cleaning is required. Some containers have preservatives present in them. Please do not rinse or overfill. Removal of some or all of the preservative may result in qualified data. Most of the chemicals used as preservatives are hazardous. Use caution when handling. Do not breathe or come in physical contact with these chemicals. For your safety, please read the enclosed Material Safety Data Sheets.

When conducting soil sampling, please clean off any residual soil from the outside of the containers. This will help prevent cross contamination of other samples in the cooler.

Please fill out all sample identification tags as completely as possible.

Please fill out the enclosed Chain of Custody form for adequate sample tracking.

The temperature requirement for the receipt of most environmental samples is  $4 \pm 2^{\circ}\text{C}$ . Temperatures that exceed this range are subject to qualification and data rejection by regulatory agencies. Following the instructions below provides the best chance of achieving and maintaining this temperature and avoiding qualified data.

- Samples should be collected and placed on ice as soon as possible. It is much more difficult to cool down warm samples.
- When possible, sample containers should be sealed in zip-lock containers. This prevents cross contamination and protects the sample labels from moisture that could render them illegible.
- Do not overfill the cooler with samples. Overfilling the cooler limits the space available for ice.
- Surround the sides and the tops of the sample containers with loose, cubed, ice. Surrounding the samples with ice is the most efficient way of cooling them. Do not use individual small bags of ice. Do not simply lay a bag of ice on top of the samples.
- Place the temperature blank in a representative location in the cooler, not in the middle of a bag of ice.
- Secure all paperwork in a zip-lock bag and place in the cooler. Seal the cooler closed.
- When shipping the coolers back to TriMatrix, complete the enclosed FedEx Airbill and attach it to the cooler. Samples shipped during the week for standard overnight delivery typically arrive the next day between 9:00 and 10:00 a.m. Saturday deliveries must be approved by your project chemist. When shipping samples for a Saturday delivery, select Priority Overnight and Saturday Delivery on the FedEx Airbill.

Please call your TriMatrix project chemist at 1-616-975-4500 if you require any further instructions, or to notify them of the pending arrival of any non-scheduled samples.

Thank You,  
TriMatrix Laboratories, Inc.





## MATERIAL SAFETY DATA SHEET

Sodium hydroxide, 50 wt% solution in water

### Section 1 - Chemical Product and Company Identification

**MSDS Name:** Sodium hydroxide, 50 wt% solution in water

**Catalog Numbers:** 25986-0000, 25986-0025, 25986-0050, 38021-0000, 38021-0025, 38021-5000

**Synonyms:** Caustic soda

**Company Identification:** Acros Organics BVBA  
Janssen Pharmaceuticaaan 3a  
2440 Geel, Belgium

**Company Identification: (USA)** Acros Organics  
One Reagent Lane  
Fair Lawn, NJ 07410

**For information in the US, call:** 800-ACROS-01

**For information in Europe, call:** +32 14 57 52 11

**Emergency Number, Europe:** +32 14 57 52 99

**Emergency Number US:** 201-796-7100

**CHEMTREC Phone Number, US:** 800-424-9300

**CHEMTREC Phone Number, Europe:** 703-527-3887

### Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	%	EINECS#	Hazard Symbols:	Risk Phrases:
1310-73-2	Sodium hydroxide	50	215-185-5	C	35
7732-18-5	Water	50	231-791-2		

Text for R-phrases: see Section 16

**Hazard Symbols:**

C





**Risk Phrases:** 35

### Section 3 - Hazards Identification

#### EMERGENCY OVERVIEW

*Causes severe burns.*

##### Potential Health Effects

**Eye:** Causes severe eye burns.

**Skin:** Causes skin burns. May cause deep, penetrating ulcers of the skin.

**Ingestion:** Causes gastrointestinal tract burns. Causes severe pain, nausea, vomiting, diarrhea, and shock. May cause corrosion and permanent tissue destruction of the esophagus and digestive tract.

**Inhalation:** Irritation may lead to chemical pneumonitis and pulmonary edema. Causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma. Causes chemical burns to the respiratory tract.

**Chronic:** Prolonged or repeated skin contact may cause dermatitis.

### Section 4 - First Aid Measures

**Eyes:** Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid immediately.

**Skin:** Get medical aid immediately. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Discard contaminated clothing in a manner which limits further exposure.

**Ingestion:** Do not induce vomiting. Get medical aid immediately.

**Inhalation:** Get medical aid immediately. Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

**Notes to Physician:** Treat symptomatically and supportively.

### Section 5 - Fire Fighting Measures

**General Information:** As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. May react with metals and lead to the formation of flammable hydrogen gas.



**Extinguishing Media:** Use foam, dry chemical, or carbon dioxide.

### Section 6 - Accidental Release Measures

**General Information:** Use proper personal protective equipment as indicated in Section 8.

**Spills/Leaks:** Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container.

### Section 7 - Handling and Storage

**Handling:** Wash thoroughly after handling. Use with adequate ventilation. Do not allow water to get into the container because of violent reaction. Do not breathe dust, vapor, mist, or gas. Do not get in eyes, on skin, or on clothing. Use only in a chemical fume hood.

**Storage:** Store in a cool, dry place. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Corrosives area. Store under an inert atmosphere.

### Section 8 - Exposure Controls, Personal Protection

#### Engineering Controls:

Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate ventilation to keep airborne concentrations low. Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits.

#### Exposure Limits

CAS# 1310-73-2:

United Kingdom, WEL - STEL: 2 mg/m<sup>3</sup> STEL

United States OSHA: 2 mg/m<sup>3</sup> TWA

Belgium - TWA: 2 mg/m<sup>3</sup> VLE

France - VME: 2 mg/m<sup>3</sup> VME

Germany: 2 mg/m<sup>3</sup> TWA (inhalable fraction)

Japan: 2 mg/m<sup>3</sup> Ceiling

Malaysia: 2 mg/m<sup>3</sup> Ceiling

Spain: 2 mg/m<sup>3</sup> VLA-EC

CAS# 7732-18-5:



**Personal Protective Equipment**

**Eyes:** Wear chemical splash goggles.

**Skin:** Wear appropriate protective gloves to prevent skin exposure.

**Clothing:** Wear appropriate protective clothing to prevent skin exposure.

**Respirators:** Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

**Section 9 - Physical and Chemical Properties**

**Physical State:** Viscous liquid

**Color:** clear colorless

**Odor:** Not available

**pH:** >13

**Vapor Pressure:** Not available

**Viscosity:** Not available

**Boiling Point:** 145 deg C ( 293.00°F)

**Freezing/Melting Point:** 12 deg C ( 53.60°F)

**Autoignition Temperature:** Not available

**Flash Point:** Not available

**Explosion Limits: Lower:** Not available

**Explosion Limits: Upper:** Not available

**Decomposition Temperature:** Not available

**Solubility in water:** Soluble

**Specific Gravity/Density:** 1.525

**Molecular Formula:** HNaO

**Molecular Weight:** 40

**Section 10 - Stability and Reactivity**

**Chemical Stability:** Stable at room temperature in closed containers under normal storage and handling conditions. Absorbs carbon dioxide from the air.

**Conditions to Avoid:** Incompatible materials, exposure to air.

**Incompatibilities with** Water, acids, aluminum, chlorinated solvents,



**Other Materials** copper, copper alloys, magnesium, phosphorus, zinc, tin, organic materials.

**Hazardous Decomposition Products** Sodium oxide.

**Hazardous Polymerization** Will not occur.

### Section 11 - Toxicological Information

**RTECS#:** CAS# 1310-73-2: WB4900000  
CAS# 7732-18-5: ZC0110000

**LD50/LC50:** RTECS:  
**CAS# 1310-73-2:** Draize test, rabbit, eye: 400 ug Mild;  
Draize test, rabbit, eye: 1% Severe;  
Draize test, rabbit, eye: 50 ug/24H Severe;  
Draize test, rabbit, eye: 1 mg/24H Severe;  
Draize test, rabbit, skin: 500 mg/24H Severe;

RTECS:  
**CAS# 7732-18-5:** Oral, rat: LD50 = >90 mL/kg;

Other:

**Carcinogenicity:** Sodium hydroxide - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.  
Water - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.

**Other:** See actual entry in RTECS for complete information.

### Section 12 - Ecological Information

Not available

### Section 13 - Disposal Considerations

Dispose of in a manner consistent with federal, state, and local regulations.

### Section 14 - Transport Information

	<b>IATA</b>	<b>IMO</b>	<b>RID/ADR</b>
<b>Shipping Name:</b>	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION
<b>Hazard Class:</b>	8	8	8
<b>UN</b>			



<b>Number:</b>	1824	1824	1824
<b>Packing Group:</b>	II	II	II

USA RQ: CAS# 1310-73-2: 1000 lb final RQ; 454 kg final RQ

## Section 15 - Regulatory Information

### European/International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols: C

Risk Phrases:

R 35 Causes severe burns.

Safety Phrases:

S 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

S 37/39 Wear suitable gloves and eye/face protection.

S 45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

WGK (Water Danger/Protection)

CAS# 1310-73-2: 1

CAS# 7732-18-5: Not available

Canada

CAS# 1310-73-2 is listed on Canada's DSL List

CAS# 7732-18-5 is listed on Canada's DSL List

### US Federal

TSCA

CAS# 1310-73-2 is listed on the TSCA Inventory.

CAS# 7732-18-5 is listed on the TSCA Inventory.

## Section 16 - Other Information

### Text for R-phrases from Section 2

R 35 Causes severe burns.

**MSDS Creation Date:** 7/16/1996

**Revision #1 Date** 5/05/2004

**Revisions were made in Sections:** General revision.

*The information above is believed to be accurate and represents the best*



*information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall the company be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential, or exemplary damages howsoever arising, even if the company has been advised of the possibility of such damages.*

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## **IMPORTANT INFORMATION FOR THE COLLECTION OF VOLATILE ORGANIC DRINKING WATER SAMPLES**

Open the water tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Reduce the water flow and carefully collect a set of duplicate samples. It is important that the flow is slow enough that no air bubbles pass through the sample as the vial is being filled. Each 40 mL vial has been pre-preserved with 25 mg of ascorbic acid preservative. Fill sample vials to just overflowing, taking care not to flush out the ascorbic acid.

Prior to sealing the set of vials, each sample must also be preserved with 1:1 hydrochloric acid. Using the supplied eyedropper and vial of HCl, carefully add 2 drops of HCl to each vial. The HCl must be added after the collection of the sample. DO NOT add the HCl to the sample vial prior to collecting the sample.

**CAUTION:** The 1:1 HCl is very acidic. Handle with care.

**NOTE:** If the sample foams vigorously when the HCl is added, discard that set of samples. Collect a new set, omitting the addition of the HCl. These samples must be flagged as "not acidified" on the chain of custody.

Seal the vials, invert, and mix for 1 minute. Verify that the sealed and mixed vial is bubble and headspace free. Sample data generated from vials received with headspace will be qualified accordingly.

The samples must be chilled to about 4° C when collected, and maintained at that temperature until analysis. Samples must be packaged for shipment with sufficient ice to ensure they arrive at the laboratory with a substantial amount of ice remaining in the cooler. Do not use Blue Ice. Surrounding the samples with crushed or cubed ice is strongly recommended. Samples received at the laboratory within 6 hours of collection may not have sufficient time to cool to 4° C. Provided that they have been correctly packed in ice, no qualifications will be necessary. Samples received in excess of 6 hours of the time of collection that exceed the required preservation temperature will be qualified accordingly.

Please call 1-616-975-4500 and speak to your project chemist if you have any questions. Thank you.





## Dissolved Sulfide Sample Collection and Preservation

To measure dissolved sulfide, insoluble matter in the sample must first be removed. This is accomplished by producing an aluminum hydroxide floc using sodium hydroxide and aluminum chloride. The flocculent is allowed to settle and the supernatant decanted off and preserved with zinc acetate. It is important that there is no headspace present in the bottle after the addition of the aluminum chloride. The vials containing the final decanted sample must also be headspace free. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

### Supplies

<i>Quantity</i>	<i>Item</i>
1 per sample	250 mL amber bottle containing 0.5 mL (10 drops) 6N NaOH
2 per sample	40 mL VOA vials, each containing 0.1 mL (2 drops) 2N Zinc Acetate per sample
2 or 3	eye droppers
1	Container of Aluminum Chloride. Enough has been sent to allow for the addition of 10 drops (0.5 mL) to each 250 mL sample.

### Procedure

- 1.0 Collect the sample in the 250 mL amber bottle containing the NaOH. Completely fill the bottle (must be enough sample so when capped it is headspace free).
- 2.0 Immediately add 10 drops of the Aluminum Chloride solution.
- 3.0 Mix the sample by holding the bottle in an upright position and rotating your wrist back and forth for 1 minute.
- 4.0 Allow the sample to settle for 5 to 15 minutes (long enough to allow the flocculent to settle to the bottom of the bottle but not longer than 15 minutes). Wait only as long as necessary to collect 80 mL of supernatant.
- 5.0 Carefully decant the supernatant into the (2) 40 mL VOA vials containing the 2N zinc acetate. Completely fill the vials with sample so they are headspace free.
- 6.0 The sample remaining in the 250 mL amber bottle is caustic. Please return the partially filled bottle to TriMatrix for disposal.





## **IMPORTANT INFORMATION FOR SULFIDE SAMPLE COLLECTION**

The amber, 500 mL, light green-tagged bottles supplied for sulfide sample collection have been pre-preserved with 1 mL of 2N zinc acetate. Sulfide samples must also be preserved with sodium hydroxide to a pH of  $\geq 9$ ; however, to correctly preserve the sulfide in the sample the addition of the sodium hydroxide must be made *after* the sample has been combined with the zinc acetate. A 4 mL vial containing 2 mL of 10N sodium hydroxide has been included with every 500 mL sulfide sample bottle for this purpose.

With a minimum of aeration, fill a 500 mL bottle up to the neck with sample. Cap and gently swirl to mix the sample and the zinc acetate. Open the sample bottle and transfer all of the sodium hydroxide from one of the 4 mL vials. Carefully add more sample to fill the 500 mL bottle, cap and mix. The filled sample container should be headspace free.

**CAUTION:** The 10N sodium hydroxide solution is very caustic. Handle with care.

Please call 1-616-975-4500 and speak to your project chemist with any questions. Thank you.





## IMPORTANT INFORMATION FOR AVAILABLE CYANIDE SAMPLE COLLECTION

Two sample containers must be collected at each sample point. One container will be treated with lead carbonate and sodium hydroxide, and the second with only sodium hydroxide (see below and the attached flowchart). A form titled "Available Cyanide Sample Treatment Record" has been provided to document all field pre-treatment activities. Please complete it as you collect and treat each sample. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

**IMPORTANT:** To avoid analyte loss it is required that all sample treatments occur within 15 minutes of sample collection.

**CAUTION:** All containers labeled as Sodium Hydroxide and Lead Carbonate/Sodium Hydroxide contain 1.3 mL of 10N sodium hydroxide. This solution is very caustic. Avoid skin contact. Handle with care.

**CAUTION:** All containers labeled as Lead Carbonate contain 0.25 g of solid lead carbonate. Avoid inhalation and skin contact.

### 1.0 Sample Collection Equipment

Per Sample

- One membrane filter
- One plastic powder funnel
- One sheet of filter paper
- One Lead Carbonate bottle
- One Lead Carbonate/Sodium Hydroxide bottle
- One Sodium Hydroxide bottle

A hand pump (not provided) is also required to perform this procedure

### 2.0 Collecting a Lead Carbonate/Sodium Hydroxide Pre-Treated Sample

If the sample contains particulates, begin with section 2.1. If the sample is particulate free, begin with section 2.2.

#### 2.1 Sample Contains Particulate Matter

If the sample contains particulate matter that would be removed upon filtration, the sample must be filtered prior to the lead carbonate pre-treatment to avoid the loss of any cyanides associated with the particulate matter. Using a powder funnel and a sheet of filter paper, filter the sample into the bottle labeled Lead Carbonate. Filter enough sample to fill the bottle up to its neck. Place the used filter paper into the bottle labeled Lead Carbonate/Sodium Hydroxide. Cap the Lead Carbonate bottle and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate



## Available Cyanide Sample Collection

---

and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate into the Lead Carbonate/Sodium Hydroxide bottle containing the used filter paper. Do not pre-rinse the container or fill to overflowing, as a loss of the particulate matter and sodium hydroxide will result. Proceed to section 3.0.

### 2.2 Sample Particulate Free

With a minimum of aeration, fill the 250 mL bottle labeled Lead Carbonate up to the neck with sample. Cap and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate collected into the bottle labeled Lead Carbonate/Sodium Hydroxide. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

### 3.0 Collecting a Sodium Hydroxide Pre-Treated Sample

With a minimum of aeration fill the 250 mL bottle labeled Sodium Hydroxide with sample. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

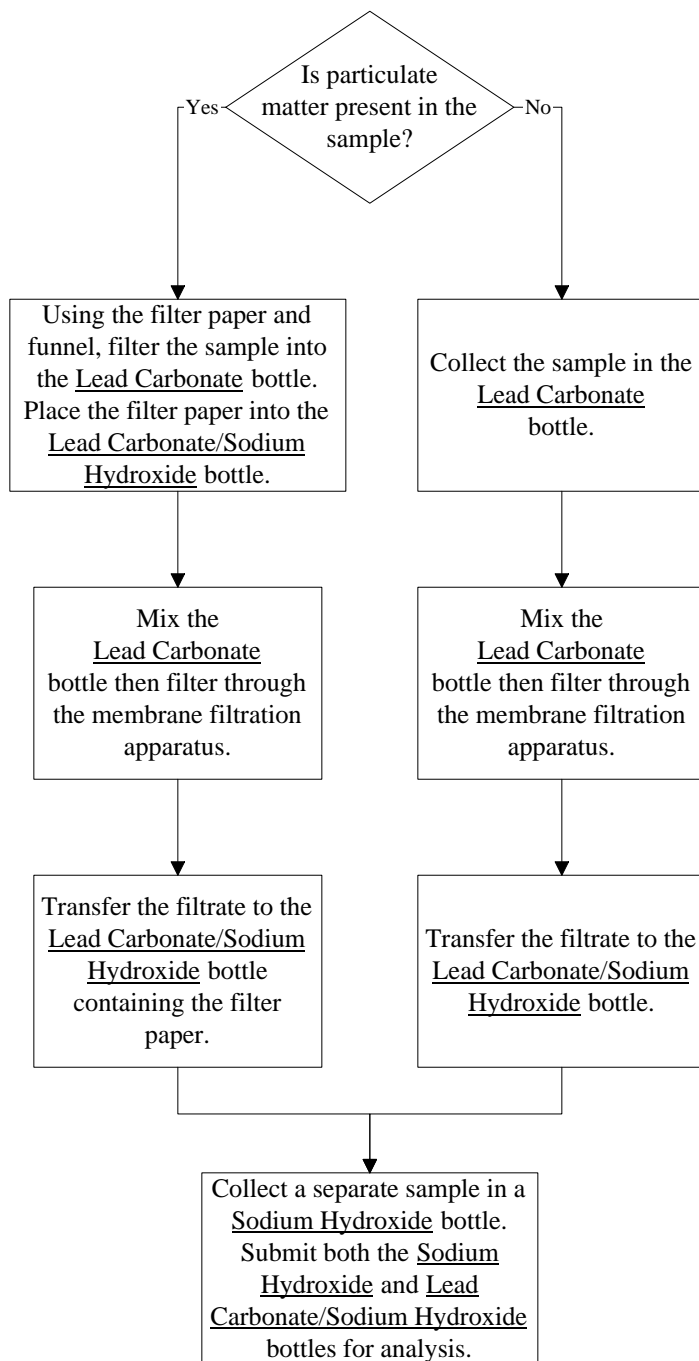
### 4.0 Collect all Paperwork and Return the Samples to TriMatrix

Place all samples in the cooler. Surround the samples with ice. To avoid data qualification all samples must be received at a temperature of between 0 and 6° C. Seal all paperwork in the resealable bag. Place the sealed bag containing the paperwork. Place all plastic powder funnels and unopened membrane filters in the cooler. Seal the cooler and return it to TriMatrix.

If you have any questions, please call TriMatrix at 1-616-975-4500 and speak with your project chemist. Thank you.



## Available Cyanide Sample Collection Flowchart





# Appendix AD





5560 Corporate Exchange Court SE Grand Rapids, MI 49512  
Phone (616) 975-4500 Fax (616) 942-7463  
www.trimatrixlabs.com

# Chain of Custody Record

COC No. \_\_\_\_\_

<b>For Lab Use Only</b>															<b>Analyses Requested</b>																
Cart																															
VOA Rack/Tray			Client Name						Project Name																						
Receipt Log No.			Address						Client Project No. / P.O. No.																						
Project Chemist									Invoice To <input checked="" type="radio"/> Client <input type="radio"/> Other (comments)																						
Work Order No.			Phone						Contact/Report To																						
			Fax																												
															Container Type (corresponds to Container Packing List)																
Schedule	Matrix Code	Laboratory Sample Number	Sample ID				Cooler ID	Sample Date	Sample Time	Comp / Grab	Matrix													Total	Sample Comments						
																		Number of Containers Submitted													
			1																												
			2																												
			3																												
			4																												
			5																												
			6																												
			7																												
			8																												
			9																												
			10																												
Sampled By (print)			How Shipped?						Comments																						
Sampler's Signature			Tracking No.																												
Company			1. Relinquished By                      Date                      Time						2. Relinquished By                      Date                      Time						3. Relinquished By                      Date                      Time																
			1. Received By                      Date                      Time						2. Received By                      Date                      Time						3. Received For Lab By                      Date                      Time																



# Appendix AE





## pH Strip Calibration Logbook

Date	Lot #	pH 4	pH 7	pH 10	Area

### pH STRIP CALIBRATION CRITERIA CORRECTIVE ACTION

1. The acceptance range for the strips is to read the exact pH of the buffer being checked. The wide range strips must pass this criteria at all three levels, 4, 7, and 10. The narrow range pH 5-7 strips are only checked at a pH of 7.
2. If the pH strips do NOT read at their appropriate levels, that lot number must NOT be used. Return them to purchasing.



# Appendix AF



## **SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS**

The collection of the sample is the starting point for the generation of quality data. It is the responsibility of TriMatrix to provide the client who collects the sample with sample collection instructions, which ensure sample integrity. Also, where applicable TriMatrix also supplies the client with appropriate clean sample containers and preservative chemicals; these glass containers are purchased new and certified as clean and vendors such as I-Chem Research and Fischer Scientific.

Sampling and Preservation Requirements for certain common environmental analyses are listed in the following table: (NOTE: Holding times are based on EPA guidelines for CLP, NPDES, and RCRA).



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
<b>ORGANICS</b>							
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015, 8021, 8260	Yellow/Black
Halocarbons*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	601	Yellow/Black
	Water	14 days	4° C/HCl to pH <2	2-40 mL VOA vials	40 mL each	601, 8015, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar	fill the jar	8015, 8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015, 8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015, 8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015, 8021, 8260	Light Yellow
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	602	Yellow/Black
Aromatics*	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	602, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8021, 8260	Light Yellow
Acrolein*	Water	3 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4° C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
Acrylonitrile*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4° C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
TPH-GRO	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015	Yellow/Black
	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	8015	Yellow
TPH-GRO/PVOC	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	Wisconsin PUBL-SW-140	Yellow
TPH-GRO	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8015	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015	Light Yellow
TPH-GRO/PVOC	Soil (Encore)	48 hours/21 days	4° C	10 or 25 g Encore	See Table 1 in Method	Wisconsin PUBL-SW-140	Label on Bag



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	Wisconsin PUBL-SW-140	Light Yellow
Petroleum	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8015	Salmon
Hydrocarbons (DRO)	Water	7 days/47 days	4° C/HCl to pH <2.0	1000 mL amber glass bottle	1000 mL	Wisconsin PUBL-SW-141	Gray
	Soil/Waste (High Level Bulk)	14 days/54 days	4° C	60 mL glass jar or	fill the jar	8015	Manila
	Soil/Waste	10 days/47 days	4° C	Tared VOC vial	See Table 1 in Method	Wisconsin PUBL-SW-141	Gray
Pesticides	Water	7 days/47 days	4° C/pH 5-9	1000 mL amber glass bottle	1000 mL	608	Yellow/White
PCBs	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	608, 8082	Salmon
Methoxychlor	Water	7 days/47 days	4° C/pH 6-8	1000 mL amber glass bottle	1000 mL	608.2	Yellow/White
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8081	Manila
PCBs	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8082	Manila
PCB Oils	Oil	N/A	None	40 mL VOA vial	20 mL	8082	Manila
Organo- phosphorous	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8141	Salmon
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8141	Manila
Phenoxy Acid Herbicides	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8151	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8151	Manila
Polynuclear aromatic	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	610, 8100	Salmon
Hydrocarbons*	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8310, 8270	Manila
Acid Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8041, 8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8041, 8270	Manila
Base/Neutral Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8270	Manila
TCLP- Volatiles	Soil/Waste	14 days/28 days	4° C	60 mL glass jar	100 g	1311	Yellow/Black
Semi-Volatiles	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Metals	Soil/Waste	180 days/360 days (Hg-28 days/56 days)	None	125 mL glass jar	250 g	1311	Manila
Pesticide/Herbicide	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Dioxins/	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	Screen-625	Salmon



**SAMPLE COLLECTION GUIDELINES**  
**BOTTLE AND PRESERVATIVE REQUIREMENTS**

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Furans	Soil/Waste	None Required	4° C	60 mL glass jar	fill the jar	Screen-625	Manila



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
<b>METALS</b>							
Metals, Total (including phosphorus)	Water	6 months	HNO <sub>3</sub> to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red
Metals, Dissolved (including phosphorus)	Water	6 months	HNO <sub>3</sub> to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red/White Stripe
	Soil/Waste	6 months	None	250 mL plastic bottle	50 g	6010/6020	White
Mercury Cold Vapor	Water	28 days	HNO <sub>3</sub> to pH <2.0	500 mL plastic bottle	500 mL	245.1, 7470	Red
	Soil/Waste	28 days	4° C	250 mL plastic bottle	50 g	7471	White
Low-Level	Water	28 days	None	500 mL borosilicate glass bottle**	500 mL	1631	Label on Bag



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
<b>INORGANICS</b>							
Color (Apparent)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Color (True)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Oil & Grease (HEM and SGT)	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	1000 mL glass bottle	1000 mL	9070/1664	Dark Blue
	Soil/Waste	28 days	None	60 mL glass jar	50 g	9071	Manila
Specific Conductance	Water	28 days	4° C	125 mL plastic bottle	100 mL	2510 B./120.1/9050	Green
Acidity	Water	14 days	4° C	125 mL plastic bottle	100 mL	2310 B.	Green
pH	Water	24 hours	4° C	125 mL plastic bottle	100 mL	150.1/9041/4500-H B.	Green
	Soil/Waste	24 hours	4° C	60 mL glass jar	50 g	9040/9041/9045	
Alkalinity	Water	14 days	4° C	125 mL plastic bottle	100 mL	310.1/2320 B.	Green
Hardness	Water	6 months	HNO <sub>3</sub> to pH <2.0	125 mL plastic bottle	100 mL	130.2/2340 C.	Red
Biochemical Oxygen Demand (BOD)	Water	48 hours	4° C	1000 mL plastic bottle	1000 mL	5210 B.	Green
Chemical Oxygen Demand (COD)	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	125 mL plastic bottle	100 mL	410.4/5220 D.	Dark Blue
Chromium (Hexavalent)	Water	24 hours	4° C	500 mL plastic bottle	500 mL	7196A, 3500-Cr B.	Green
	Soil/Waste	30 days/24 hours	4° C	60 mL glass jar	50 g	7196A	Manila
Organic Carbon (TOC)	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	3-40 mL VOA vials	40 mL	415.1/5310 D./9060	Salmon
	Soil/Waste	28 days	4° C	60 mL glass jar	10 g	MSA 29-3.5.2/415.1/9060	Manila



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Ortho- Phosphate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	365.1/4500-P E.	Green
Total Phosphorus	Water	28 days	H <sub>2</sub> SO <sub>4</sub> to pH <2.0	125 mL plastic bottle	100 mL	365.1/4500-P F.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	365.1/4500-P F.	Manila
Total Kjeldahl Nitrogen (TKN)	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	125 mL plastic bottle	100 mL	351.2	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	351.2	Manila
Ammonia	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	125 mL plastic bottle (500 mL for wastewater)	100 mL (200 mL for wastewater)	350.1/4500-NH <sub>3</sub> G.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	350.1/4500-NH <sub>3</sub> G.	Manila
Nitrite	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/354.1/ 4500 NO <sub>2</sub> -B/4500 NO <sub>2</sub> -F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	353.2/9056	Manila
Nitrate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/4500 NO <sub>3</sub> -F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	9056/353.2/4500 NO <sub>3</sub> -F	Manila
Nitrite plus Nitrate (No distinction between NO <sub>2</sub> and NO <sub>3</sub> )	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	125 mL plastic bottle	100 mL	353.2/4500 NO <sub>3</sub> -F	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	353.2/4500 NO <sub>3</sub> -F	Manila
Total Volatile Solids	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.4	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	2540-G	Manila
Turbidity	Water	48 hours	4° C	125 mL plastic bottle	100 mL	180.1/2130 B.	Green
Sulfate	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/375.4/9038	Green
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	9056/375.2/9038/4500 SO <sub>4</sub> -F	Manila
Sulfite	Water	48 hours	4° C/3 mL 1% EDTA	125 mL plastic bottle	100 mL	377.1	Manila



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Sulfide, Total	Water	7 days	4° C/Pre-Preserved with Zinc Acetate; NaOH Added in field to pH ≥9	125 mL plastic bottle	100 mL	9034/376.1/376.2/4500 S <sub>2</sub> -D 4500 S <sub>2</sub> -F	Light Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	9034	Manila
Cyanide*	Water	14 days	4° C/NaOH to pH >12	1000 mL plastic bottle	1000 mL	335.2/335.4/9012/9014	Light Blue
	Soil/Waste	14 days	4° C	60 mL glass jar	50 g	9012/9014	Manila
Cyanide, Available	Water	14 days	1 Lead Carbonate bottle 1 Lead Carbamate/NaOH bottle 1 NaOH bottle	125 mL amber glass bottles	125 mL	OIA-1677	Light Blue
Coliform Fecal and Total	Water	24 hours	4° C/Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	Sterile plastic bottle or Whirl-Pak	100 mL	9222-D/9223-B	White
Bromide	Water	28 days	4° C	125 mL plastic bottle	100 mL	9056/ASTM D1246-88	Green
Chloride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/325.2/4500-Cl E.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056/325.2/4500-Cl E.	Manila
Chlorine Residual	Water	Analyze Immediately	4° C	125 mL plastic bottle	100 mL	HACH-8167	Green
Total Solids (% Moisture)	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.3/2540 B.	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	3550	Manila
Total Dissolved Solids (TDS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.1/2540 C.	Green
Total Suspended Solids (TSS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.2/2540 D.	Green
Fluoride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/4500-F C.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056	Manila



## SAMPLE COLLECTION GUIDELINES BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Organic Halogen (TOX)	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	500 mL amber glass bottle	500 mL	9020	Lilac
	Soil	28 days	4° C	60 mL glass jar	50 g	9023	Manila
Phenolics	Water	28 days	4° C/H <sub>2</sub> SO <sub>4</sub> to pH <2.0	500 mL amber glass bottle	100 mL	420.2/420.4/9066	Brown
	Soil	28 days	4° C	60 mL glass jar	50 g	9066	Manila
Surfactants (MBAS)	Water	48 hours	4° C	1000 mL plastic bottle	400 mL	425.1/5540 C.	Green
Flash Point	Solid/Liquid/Waste	N/A	None	Clear glass wide mouth jar. 60 mL unless otherwise specified.	100 g	1010/1020	White
	Waste	N/A	None		100 g	1010/1020	White
Corrosivity (pH and Method 1110)	Waste	N/A	None	(Appropriate to Sample) 500 mL glass or plastic bottle	500 mL	9040/9041/1110	White
Paint Filter (Free Liquids)	Soil/Waste	N/A	None	(Appropriate to Sample) 250 mL glass jar or 125 mL plastic bottle	100 g	9095	White
Radiologicals (Alpha + Beta, Alpha, Beta, Ra 226, Ra 228)	Water	6 months	HNO <sub>3</sub> to pH <2.0	1000 mL plastic bottles or 1000 mL glass bottle	1000 mL		White
Reactivity (Releasable CN and S)	Waste	14 days CN, 7 days S	4° C	(Appropriate to Sample) 125 mL plastic bottle or 60 mL glass jar	10 g	SW- 846 Chapter 7	White

\*Sample must also be preserved with Sodium Thiosulfate or Ascorbic Acid if chlorinated

\*\*All low-level mercury bottles are stored filled with 5 mL of concentrated HCl and Millipore water

NOTE: For Organics parameters, container lid should be Teflon.

NOTE: For Inorganic parameters, container lid should be plastic or Teflon lined.

NOTE: When testing for several like parameters (ICP metals, Ion Chromatograph anions), one container per sample is sufficient. For example, a sample to be tested for the 13 priority pollutant metals needs one 500 mL container.



# Appendix AG



Client: **S**  
Project: **Analytical Services**

Project Manager: **Jennifer L. Rice**  
Date Received: **Dec-08-08 10:00**

Department: **Metals**

Analysis: \_\_\_\_\_

<i>Lab Number / Sample Name</i>	<i>Container</i>	<i>Removed by (Signature)</i>	<i>Date &amp; Time Removed</i>	<i>Date &amp; Time Returned</i>	<i>Consumed?</i>	<i>Extract Container</i>
<b>0812144-01</b> Pugged CKD	_____	_____	_____	_____	_____	_____
<b>0812144-02</b> Pugged CKD	_____	_____	_____	_____	_____	_____
<b>0812144-03</b> Pugged CKD	_____	_____	_____	_____	_____	_____
<b>0812144-04</b> Pugged CKD	_____	_____	_____	_____	_____	_____



# Appendix AH





**TriMatrix**  
Laboratories, Inc.

## *Non-Conformance Investigation Report*

Client: \_\_\_\_\_ Project Number: \_\_\_\_\_

Sample Number(s): \_\_\_\_\_ Date Initiated: \_\_\_\_\_ Date Due: \_\_\_\_\_

Initiated By: \_\_\_\_\_ Document Control Number: \_\_\_\_\_

Investigation Resulting From: ☐ Internal Observation ☒ Client Complaint ☐ Audit ☐ Failing PT Sample

### *I. Area of Non-Conformance:*

☐ Sample Receiving / Storage ☐ Bottle Prep ☐ Client Services / Reporting ☐ Other \_\_\_\_\_

☒ Inorganic (**Wet Chemistry** / Metals) Laboratory ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

### *II. Description of Non-Conformance:*

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### *III. Explanation of Investigation into Non-Conformance:*

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Initials: \_\_\_\_\_ Date: \_\_\_\_\_

### *IV. Resolution:*

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Initials: \_\_\_\_\_ Date: \_\_\_\_\_

### *V. Follow-Up (if required):*

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Initials: \_\_\_\_\_ Date: \_\_\_\_\_

### *VI. Reviewed By:*

QA Manager: \_\_\_\_\_ Area Manager: \_\_\_\_\_

Date Completed: \_\_\_\_\_



# Appendix AI





**TriMatrix**  
Laboratories, Inc.

### Preventive Action Investigation

**Initiated By:** \_\_\_\_\_ **Document Control Number:** \_\_\_\_\_

**Date Initiated:** \_\_\_\_\_ **Date Due:** \_\_\_\_\_

Investigation Resulting From: ☐ Internal Observation ☐ Client Complaint ☐ Audit ☐ Failing PE Sample

***I. Area of Preventive Action:***

☐ Sample Receiving / Storage    ☐ Bottle Prep    ☐ Client Services / Reporting    ☐ Other\_\_\_\_\_

☐ Inorganic (Wet Chemistry / Metals) Laboratory      ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

## II. Description and Proposed Solutions:

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### III. Action Plan and Implementation Schedule:

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### V. Follow-Up to Monitor Effectiveness:

Initials: \_\_\_\_\_ Date: \_\_\_\_\_

**VI. Reviewed By:**

**QA Manager:** \_\_\_\_\_ **Area Manager:** \_\_\_\_\_

Date Completed: \_\_\_\_\_



## **8.0 GLOSSARY OF TERMS**

**ABSORBANCE** - a measure of the decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \left( \frac{I(\text{solvent})}{I(\text{solution})} \right) - \frac{\log I_0}{I}$$

**ALiquot** - a measured portion of a field sample taken for analysis.

**ANALYSIS DATE/TIME** - the date and time of the introduction of the sample, standard, or blank into the analysis system.

**ANALYTE** - the element or ion an analysis seeks to determine; the component of interest.

**ANALYTICAL SAMPLE** - any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRDL standard for AA (CRA), CRDL standard for ICP (CRI), laboratory control sample (LCS), method preparation blank (MPB), laboratory fortified blank (LFB), and linear range analysis sample (LRS).

**AUTOZERO** - zeroing the instrument at the proper wavelength. It is equivalent to running a blank to set the absorbance to zero.

**AVERAGE INTENSITY** - the average of two different responses from a detector.

**BACKGROUND CORRECTION** - a technique to compensate for background contribution to the instrument signal in the determination.



**BLANK** - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

**BATCH** - a group of samples prepared at the same time in the same location using the same method.

**BREAKDOWN** - a measure of the decomposition of certain analytes (i.e. DDT and Endrin) into by-products.

**4-BROMOFLUOROBENZENE (BFB)** - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

**CALIBRATION** - the establishment of an analytical curve based on the measured response of known standards.

**CALIBRATION BLANK** - a volume of laboratory reagent or other inert carrier matrix.

**CALIBRATION STANDARDS** - a series of known standards used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

**CALIBRATION FACTOR (CF)** - a measure of the gas chromatographic response of a target analyte to the mass injected during external calibration. The calibration factor is analogous to the Response Factor (RF) calculated during internal calibration.

**CASE** - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

**CONTAMINATION** - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.



**CONTINUING CALIBRATION VERIFICATION** - analytical standard run at periodic intervals to verify the initial calibration of the system.

**CONTRACT REQUIRED DETECTION LIMIT (CRDL)** - minimum level of detection acceptable as specified by the project to report.

**CONTROL LIMITS** - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

**CORRELATION COEFFICIENT** - the number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the number (r) is. Determined on the basis of the least squares regression.

**DAY** - unless otherwise specified, day shall mean calendar day.

**DIGESTION LOG** - an official record of the sample preparation (digestion).

**DISSOLVED METALS** - analyte elements which have not been digested prior to analysis and which will pass through a 0.45 um filter.

**DRY WEIGHT** - the weight of a sample analyzed based on percent solids. The weight after drying in an oven.

**DUPLICATE** - a second aliquot of sample that is treated the same as the original in order to determine the precision of the collection.

**EXTRACTED ION CURRENT PROFILE (EICP)** - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

**EXTRACTABLE** - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.



**FIELD BLANK** - any sample submitted from the field identified as a blank.

**FIELD SAMPLE** - Material received to be analyzed that is contained in single or multiple containers and identified by a unique Sample Number.

**GAS CHROMATOGRAPH (GC)** - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are either volatilized directly from the sample (VOA water and low-soil), from the sample extract (VOA medium soil), or injected as an extracted sample (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture Detector (ECD). In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

**HOLD TIME** - the maximum allowable elapsed time expressed in hours or days from the time the sample is collected until the time of its pre-treatment or analysis.

**INDEPENDENT STANDARD** – an externally prepared standard solution composed of analytes from a different source than those used in the standards for the initial calibration.

**INDUCTIVELY COUPLED PLASMA (ICP)** - a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

**IN-HOUSE** - at the laboratories facility.

**INITIAL CALIBRATION** - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the instrument.

**INJECTION** - introduction of the analytical sample into the instrument excitation system for the purpose of measuring concentration of an analyte.



**INSTRUMENT CALIBRATION** - Series of analytical standards at different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument.

**INSTRUMENT DETECTION LIMIT (IDL)** - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

**INSTRUMENT CHECK SAMPLE** - a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

**INSTRUMENT CHECK STANDARD** - a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

**INTERFERENTS** - substances which affect the analysis for the element of interest.

**INTERNAL STANDARDS** - compounds added to analytical and quality control samples at a known concentration prior to analysis. In the methods that require them, internal standards are used as the basis for quantitation of the target compounds.

**INSTRUMENT/ANALYTICAL BLANK** - a blank designed to determine the level of contamination associated with the analytical instrument.

**INSUFFICIENT QUANTITY** - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

**SECOND SOURCE CALIBRATION VERIFICATION (SCV) STANDARD** - a standard prepared from a source other than that used to prepare the quantitation standard, and used to verify the initial calibration curve.



**BLANK SPIKE** - a control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

**LABORATORY RECEIPT DATE** - the date on which a sample is received as recorded on the chain of custody.

**LINEAR RANGE, LINEAR DYNAMIC RANGE** - the concentration range over which the determinative instrument's analytical curve remains linear.

**MATRIX** - the predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

**MATRIX EFFECT** - in general, the effect of the particular sample matrix on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

**MATRIX SPIKE** - aliquot of a matrix spiked with known quantities of target compounds and subjected to the entire analytical procedure. Matrix spikes are used to indicate the efficiency of the method on the matrix by measuring the recovery of the spiked analyte.

**MATRIX SPIKE DUPLICATE** - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method relative to the matrix.

**METHOD BLANK** - an analytical control consisting of all reagents, internal standards and surrogate standards that are carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

**METHOD OF STANDARD ADDITIONS (MSA)** - the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept.



Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix effects; it will not counteract special effects. Also referred to as Standard Addition.

m/z - Mass to charge ratio, synonymous with "m/e"

NARRATIVE - portion of the data package which includes laboratory, contract, case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference).

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105° C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105° C, including water.

PERCENT SOLIDS - the proportion of solid in a soil sample determined by drying an aliquot of the sample at 105° C.

PERFORMANCE EVALUATION MIXTURE - a calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE TESTING (PT) SAMPLE - a single blind sample of known composition obtained from an external provider for analysis. Used by clients and regulatory agencies to evaluate laboratory performance.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled/deionized water and reagents, which is carried through the entire analytical procedure – digested/distilled/extracted and analyzed. An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.



**PRIMARY QUANTITATION ION** - a specific ion used to quantitate a target analyte.

**PROTOCOL** - a compilation of procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

**PURGE AND TRAP (DEVICE)** - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto a gas chromatographic column.

**PURGEABLES** – non-water soluble volatile organic compounds.

**QUALITY CONTROL SAMPLE** - a solution obtained from an outside source having known concentration values to be used to verify the calibration.

**REAGENT BLANK** - a volume of deionized, distilled water containing the same reagent matrix as the calibration standards carried through the entire analytical scheme.

**REAGENT WATER** - water in which an interferent is not observed at or above the minimum detection limit of the parameters of interest.

**RECONSTRUCTED ION CHROMATOGRAM (RIC)** - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

**RELATIVE PERCENT DIFFERENCE (RPD)** - The relative percent difference is based on the mean of two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

**RELATIVE RETENTION TIME (RRT)** - the ratio of the retention time of a compound to that of a standard (such as an internal standard).



$$RRT = \frac{RT_c}{RT_{is}}$$

where,

RT<sub>c</sub> = Retention time for the target or surrogate compound in continuing calibration.

RT<sub>is</sub> = Retention time for the internal standard in calibration standard or in a sample.

**RELATIVE STANDARD DEVIATION (RSD)** - the variation of a series of results based on the standard deviation and average. Typically used in the evaluation of initial calibration curves.

$$RSD = \frac{SD}{\text{Average RF}}$$

**RESOLUTION** - the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

**RESPONSE** - or Instrumental Response: a measurement of the output of the detector in which the intensity of the signal is proportionate to the concentration detected.

**RESPONSE FACTOR (RF)** - a measure of the relative response of an analyte compared to an internal standard. The RF is determined by the following equation:

$$RF = \left( \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \right)$$

where:

A = area of the characteristic ion measured

C = concentration

is = internal standard

x = analyte of interest

**RETENTION TIME (RT)** - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is



dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

**ROUNDING RULES** - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no known figures beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

**RUN** - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements.

**SAMPLE** - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

**SAMPLE NUMBER** - a unique identification number designated for each sample. The Sample Number appears on all laboratory documents which contain information on that sample.

**SEMIVOLATILE COMPOUNDS** - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

**SENSITIVITY** - the slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.



**SERIAL DILUTION** – a series of dilutions to attain a less concentrated solution.

**SOIL** - synonymous with soil/sediment or sediment as used herein.

**SONICATOR** - a device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given solid matrix.

**SPECTRA** - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

**STORAGE BLANK** - a reagent water aliquot stored with samples and analyzed on a weekly basis for VOCs. The storage blank is used to determine the potential for sample contamination occurring during storage.

**STOCK SOLUTION** - a standard solution prepared from neat materials diluted to derive other standards.

**SURROGATES (Surrogate Standard)** - for semivolatiles, volatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be present in the sample.

**SUSPENDED** - those particulates in suspension which are retained by a 0.45 um membrane filter.

**TENTATIVELY IDENTIFIED COMPOUNDS (TIC)** - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

**TOTAL METALS** – analytes from the sample which have been digested to complete solvency prior to analysis.

**TWELVE-HOUR TIME PERIOD** - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the



laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. The injection time of the last analyses in the batch must be made within 12 hours of the injection time of BFB of DFTPP.

**VOLATILE COMPOUNDS** – non-water soluble compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

**WET WEIGHT** - the mass of a sample aliquot including moisture (un-dried) that is used for analysis.

**WIDE BORE CAPILLARY COLUMN** - a gas chromatographic column with an internal diameter (ID) that is greater than 0.32 mm. Columns with lesser diameters are classified as capillary columns.



## **Attachment T-1**

Laboratory Standard Operating  
Procedures





**TRIMATRIX**  
LABORATORIES

## STANDARD OPERATING PROCEDURE

### Mercury by semi-Automated Cold Vapor Atomic Absorption

**EPA Method 245.1**  
**SW-846 Method 7470A**  
**SW-846 Method 7471A**

#### APPROVALS:

Area Supervisor: \_\_\_\_\_

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Denise S. Coffey

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*4-6-10*

QA Officer: \_\_\_\_\_

*Tom C. Boocher*  
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Date: \_\_\_\_\_

*4/11/10*

Procedure Number: GR-01-123  
Revision Number: 5.7

Date Initiated: 5/6/93

Effective Date: 4/15/10

Date Revised: 4/2/10

Pages Revised: All

By: David W. Johnson  
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed

Reviewed by

Review Expires

_____	_____	_____
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## 1.0 SCOPE AND APPLICATION

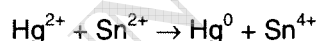
- 1.1 This procedure includes analysis for total mercury (inorganic/organic) in mobility extracts, aqueous wastes, groundwaters, drinking waters, surface waters, saline waters, wastewaters, domestic industrial wastes, soils, wastes, sludges and oils. All samples must undergo a preliminary digestion.
- 1.2 The typical reporting limit is 0.2 ug/L mercury for aqueous and 0.05 mg/kg for solid sample matrices.

## 2.0 PRINCIPAL METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update II, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"*
- 2.2 *Methods for the Determination of Metals in Environmental Samples, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994*
- 2.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update II, Revision I, September 1994, Method 7471A, "Mercury in Solid and Semi Solid Waste (Manual Cold-Vapor Technique)"*

## 3.0 SUMMARY OF PROCEDURE

- 3.1 Before analysis, all analytical and quality control samples must be pretreated according to the references discussed in Section 5.0. These digestions reduce organo-mercury compounds to inorganic mercury. Inorganic mercury is then converted to mercury in the  $Hg^{2+}$  state. All  $Hg^{2+}$  with an HCl carrier enters a gas-liquid separatory where it is mixed with stannous chloride to form elemental mercury vapor ( $Hg^0$ ) according to the following equation:



- 3.2 The mixture then flows into a liquid-gas separator where argon is introduced to carry the mercury vapor through a drying tube. The dried mercury vapor then enters one path of a heated double-path optical cell that has been optimized for fast response time. A mercury source powered by a constant current power supply delivers a stable source of absorbance at 253.7 nm. Absorbance by the mercury vapor is measured using a solid-state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.
- 3.3 Absorbance of standards is plotted against known concentrations to build a calibration curve. Absorbance of unknown samples are read against the calibration curve and regressed to

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QA Officer	Area Supervisor





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concentration in ug/L. The mercury vapor is vented up a hood to minimize contamination of other samples.

#### 4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

#### 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-01-140, *Preparation Procedure for Mercury in Water, Wastewater, and Liquid Wastes*, latest revision
- 5.3 TriMatrix SOP GR-01-139, *Preparation Procedure for Mercury in Soils, Wastes, and Oils*, latest revision
- 5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision.
- 5.5 TriMatrix SOP GR-10-123, *Element Data Transfer and Review*, latest revision
- 5.6 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision
- 5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision
- 5.8 TriMatrix SOP GR-09-128, *Homogenization, Grinding and Drying of Solid Samples*, latest revision
- 5.9 *PSA 10.045 Millenium Merlin/Galahad System User Manual*, Issue Number 1.1, Issue Date July 10, 1999

#### 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil samples often contain mercury at low levels. When soils are mixed and ground, there exists the possibility that liquid samples may become contaminated by dust from the air. Therefore, preparation areas for mercury must be kept free of dust and dirt.

Note: Mixing and grinding samples must be done in a fume hood for safety.

6.2 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory so precautionary measures against contamination can be taken.

6.3 Mercury may be lost if the digestion temperature goes above 95° C. The digestion temperature must be monitored and recorded with a digital thermometer on the digestion benchsheet.

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- 6.4 Reagents used may become contaminated over time. If contamination is suspected, the reagent must be discarded following proper laboratory disposal guidelines and fresh reagent made.
- 6.5 Potassium permanganate is added to eliminate possible interference from sulfide as sodium sulfide.
- 6.6 Copper interferes at concentrations above 10 mg/L.
- 6.7 Samples high in chlorides and volatile organic materials require additional permanganate.

Note: The maximum permanganate added to a sample in the digestion batch must be added to all associated quality control samples.

## 7.0 SAFETY PRECAUTIONS

- 7.1 Comply with all instructions for health and safety as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.
- 7.2 Concentrated acids are used. Disposable gloves, safety glasses and a laboratory coat must be worn at all times when handling concentrated acids. Gloves, safety glasses and laboratory coats must also be worn when handling digested samples or performing any work within the metals laboratory. Refer to the MSDS located on the library drive for information on any chemical used.
- 7.3 Make sure the venting system is hooked up and running. Do not operate the instrument if it is not properly ventilated.
- 7.4 Mercury lamps emit UV radiation. Avoid looking directly at the lamp without some type of strong UV protection. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.
- 7.5 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Mercury compounds can also be absorbed through the skin. Wear disposable gloves at all times. Elemental  $\text{Hg}^0$  and  $\text{Hg}^{2+}$  are toxic if ingested. Always wash hands after handling mercury standards, samples or reagents, and when leaving the laboratory.
- 7.6 No food or drink is allowed in the metals laboratory. Food and drink may be consumed in the laboratory office but not stored there.
- 7.7 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a third party through casual contact.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

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Approved By: DSC 4-6-10  
Area Supervisor




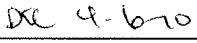
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- 8.1 Samples should be acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, lower to pH <2 during the sample receipt process. A minimum of 24 hours must elapse after pH adjustment by the laboratory before performing the sample digestion.
- 8.2 Store non-aqueous and solid samples at 0 - 6° C.
- 8.3 The maximum holding time for all mercury samples is 28 days from the collection date. Analyze non-aqueous samples as soon as possible within that time.
- 8.4 All samples must be subjected to digestion before analysis.
- 8.5 The minimum collected aqueous sample volume required is 100 mL. If quality control is also required, collect at least 300 mL. The minimum sample size for solid samples is 10 g.

## 9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Laboratory analytical balance capable of weighing 0.0001 g
- 9.2 Laboratory balance capable of weighing 0.1 g
- 9.3 Macropipettor capable of delivering 50 - 1000 µL
- 9.4 Class A volumetric flasks, various volumes
- 9.5 Class A reusable pipettes, various volumes
- 9.6 Macropipettes capable of delivering 5 - 10 mL of solution
- 9.7 Wooden laboratory spatulas
- 9.8 Calibrated digital thermometer capable of reading to 100° C. Mercury thermometers must never be used in this procedure.
- 9.9 PSA low-level absorbance mercury analyzer, Millenium system, model 10.045
- 9.10 Computer running Avalon software. An IBM compatible computer running Windows NT 4.0 service pack 5.0. The software version may be updated without notice if it performs at least as well as the older version.
  - 9.10.1 Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.
  - 9.10.2 Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

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9.11 Filter, 0.45 µm glass fiber

## 10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Check the calibration on autopipettors daily. Remove from service and send out for recertification when daily checks indicate unacceptable accuracy or performance. Perform an annual inspection to determine overall condition.

10.2 Verify balance calibration daily. Remove from service and call for repair when daily checks indicate unacceptable accuracy or performance. Recertify annually through a contracted service representative.

10.3 Inspect the waste bottle under the instrument before starting. If the bottle is full, properly dispose of the liquid.

10.4 Under normal conditions, the PSA requires very little maintenance. However, attention to the maintenance schedule below must be followed:

### 10.4.1 Daily:

10.4.1.1 Before shutting down, flush all tubing and the sample valve with laboratory reagent water. Finally, flush with air.

10.4.1.2 It may necessary to replace all tubing. However, flushing of the sample valve must still take place.

10.4.1.3 Replace the pump tubing if required. When in doubt, replace it.

10.4.1.4 Ensure that glassware is clean. When in doubt, clean it.

10.4.1.5 Clean all outer surfaces of the instrument.

10.4.1.6 Also after shutting down, release any tension on the pump tubing.

### 10.4.2 Weekly:

10.4.2.1 Perform daily maintenance activities.

10.4.2.2 Replace all pump tubing if not already replaced as part of the daily maintenance.

### 10.4.3 Monthly:

10.4.3.1 Perform daily and weekly maintenance activities.

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- 10.4.3.2 Replace all pump tubing if not already replaced as part of daily/weekly maintenance.
  - 10.4.3.3 Check for discoloration in the Perma-pure drying system and replace as necessary.
  - 10.4.4 Six Months:
    - 10.4.4.1 Perform daily, weekly and monthly maintenance.
    - 10.4.4.2 Replace the inner membrane of the Perma-pure drying system if needed.
  - 10.4.5 Yearly:
    - 10.4.5.1 Perform daily, weekly, monthly and six month maintenance.
    - 10.4.5.2 Change the inner membrane of the Perma-pure drying system if needed.
    - 10.4.5.3 Replace the activated charcoal in the exhaust line.
  - 10.4.6 Cleaning:
    - 10.4.6.1 Before undertaking any cleaning, disconnect the main supply cable from the instrument. Cleaning is ONLY recommended for the outer case.
    - 10.4.6.2 Clean with a damp cloth or sponge which has been previously immersed in warm soapy water. Alternatively, a suitable laboratory cleaning agent may be used. Avoid copious amounts of liquid coming into contact with the instrument.
    - 10.4.6.3 Finish off by wiping down with a dry lint-free cloth or sponge.
  - 10.4.7 Wipe acid spills up with a suitable neutralizing agent IMMEDIATELY. However, be careful to avoid a copious volume of liquid coming into contact with the instrument.
- 11.0 CHEMICALS AND REAGENTS**
- 11.1 Acids used in standards preparation and for samples must be trace metal grade or better.
    - 11.1.1 Concentrated hydrochloric acid, trace metal grade
    - 11.1.2 Concentrated nitric acid, trace metal grade
  - 11.2 Laboratory Reagent Water from the MilliQ System.
  - 11.3 Argon gas supply

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11.3.1 Welding grade or better. This is plumbed from a liquid argon tank located outside the building. Argon is used as the carrier gas during analysis.

11.4 Stannous chloride, 2% solution in 10% hydrochloric Acid.

11.4.1 Place 20 g stannous chloride into a clean 1 L polyethylene bottle. Add 100 mL concentrated HCl and swirl until the SnCl is dissolved. Add 880 mL ASTM Type II water then cap and mix.

11.4.2 Create a new standard number in Element™ when the stannous chloride lot changes.

## 12.0 STANDARDS PREPARATION

12.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140, for the preparation of mercury standards.

## 13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of samples by matrix.

## 14.0 CALIBRATION PROCEDURES

14.1 A blank and five standards comprise the initial calibration curve. Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of calibration standards.

14.2 Prepare a digested calibration curve the same day as digestates and use for quantitation.

## 15.0 ANALYTICAL PROCEDURE


15.1 Analysts must be familiar with the software's operation by reading the software manual and through documented training from an experienced analyst before proceeding.

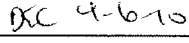
15.2 After running the calibration curve, analyze a continuing calibration verification (CCV). The CCV is prepared at 2.0 ug/L and recovery must be within 95 – 105% of the expected value for method reference 245.1 or within 90 – 110% for method references 7470A and 7471A.

15.2.1 If the CCV fails recovery, repeat the analysis.

15.2.2 If the second analysis fails, initiate corrective action:

15.2.2.1 Adjust the gas flow if needed.

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Area Supervisor





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- 15.2.2.2 Replace tubing if needed.
- 15.2.2.3 Check for other mechanical problems to verify the instrument is operating correctly.
- 15.2.2.4 Remake and redigest the calibration standards if necessary.
- 15.2.2.5 Remake the CCV standard if necessary.
- 15.2.3 After resolving the problem, recalibrate the instrument and run the CCV successfully.
- 15.2.4 Digestions may not be analyzed without an acceptable CCV.
- 15.3 Analyze a second-source calibration verification (SCV) as a sample. The SCV is prepared from a second-source purchased standard and prepared at 3.0 ug/L. SCV recovery must be within 90 – 110% .
  - 15.3.1 If the SCV fails recovery, repeat the analysis.
  - 15.3.2 If the second analysis fails, initiate corrective action:
    - 15.3.2.1 Check to make sure the instrument is operating correctly.
    - 15.3.2.2 Recalibrate.
  - 15.2.3 After resolving the problem, recalibrate the instrument and run the SCV successfully.
  - 15.2.4 Digestions may not be analyzed without an acceptable SCV.
- 15.4 Analyze a calibration blank (CCB) after the SCV. The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.
  - 15.4.1 If the CCB is higher than the reporting limit, reanalyze one time.
  - 15.4.2 If the second analysis fails, initiate corrective action.
    - 15.4.2.1 Locate the source of the contamination.
    - 15.4.2.2 Correct the contamination problem.
    - 15.4.2.3 Repeat all associated sample digestions associated with the contamination problem and/or narrate the contamination.
- 15.5 Analyze a contract-required limit (CRL) standard at the beginning of each run.
  - 15.5.1 A CRL is reanalysis of the digested 0.2 ug/L standard from the initial calibration curve as a sample.

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QA Officer	Area Supervisor



SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption  
EPA Method 245.1, SW-846 Method 7470A, SW-846 Method 7471A  
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- 15.5.2 CRL recovery must fall within 50 – 150% of the expected value. If the CRL result is above the calculated MDL value but outside the control limits, narrate in the analytical batch as follows:
- 15.5.2.1 The CRL recovery for this analyte was outside laboratory control limits. However, the only requirement is that the analyte be detected. No qualifications are necessary.
- 15.6 After analysis of the CRL, analyze a continuing calibration verification (CCV) standard. The CCV is the midpoint calibration standard analyzed as a sample. CCV recovery must be within 90 – 110% of the expected value.
- 15.6.1 If the CCV fails, reanalyze one time.
- 15.6.2 If the CCV still fails, stop the analysis and initiate corrective action.
- 15.6.2.1 Check to make sure the instrument is operating correctly.
- 15.6.2.2 Recalibrate.
- 15.6.2.3 Reanalyze all digestates since the last acceptable CCV.
- 15.7 Analyze a continuing calibration blank (CCB). The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.
- 15.7.1 If the CCB fails, reanalyze one time.
- 15.7.2 If the second analysis fails, initiate corrective action:
- 15.7.2.1 If a high level sample is carrying over, analyze the CCB until the system is clean then reanalyze all digestates run since the last acceptable CCB.
- 15.7.2.2 If no apparent cause can be found, recalibrate then reanalyze all digestates run since the last acceptable CCB.
- 15.8 After the initial quality control has passed, analyze up to 10 samples (including the high and low concentration blank spikes) then repeat analysis of the CCV and CCB. Repeat until all samples are analyzed.
- Note: Shake all digestates before analysis. Solid matrix digestates must be filtered through a 0.45 µm filter before analysis.
- 15.9 Check all raw data to make sure each result is within the calibration range. If any result is above the calibration range, mark the sample number and estimate a dilution factor. Make the dilution using a digestion CCB that matches the sample reagent matrix then analyze within the calibration range.

Approved By:           *JB* 4-2-10            
QA Officer

Approved By:           *OK* 4-6-10            
Area Supervisor



SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption EPA Method 245.1, SW-846 Method 7470A, SW-846 Method 7471A SOP Number: <b>GR-01-123</b>	Revision Number: 5.7 Date Revised: 4/2/10 Date Initiated: 5/6/93
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15.9 Verify the calibration after all samples have been analyzed as follows:

15.9.1 Analyze a final CCV successfully.

15.9.2 Analyze a final CCB successfully, which concludes the run.

## 16.0 CALCULATIONS AND DATA HANDLING

16.1 Collect data from the instrument and transfer to the laboratory information management system in accordance with TriMatrix SOP GR-10-123.

16.2 Duplicate measurements are taken and recorded for each sample. The average of these two readings is reported as the final result.

## 17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for correctly filling in, handing in and filing all associated paperwork. It is essential to perform these tasks for the laboratory to provide clients with defensible data.

### 17.2 LIMS Reporting

17.2.1 When finished running a sample set, all data must be uploaded to Element™, the Laboratory Information Management System (LIMS) in accordance with TriMatrix SOP GR-10-123.

17.2.1.1 To export a data set from the instrument for LIMS uploading with DataTool, click on the red arrow.

17.2.1.2 Choose "comma" and "browse" then select the "groups\lab.me\export data\216" folder and click "OK".

17.2.1.3 Add a backslash (\) and type the data set filename (include ".res" on the end). For example, to export dataset y17wta2, the path appears as "groups\lab\_me\exportdata\216\y17wta2.res".

17.2.1.4 Click "OK" to export the data.

17.2.2 If an internal chain-of-custody is required, it is very important that COC forms be filled in correctly and returned to the COC file location. Refer to Attachment 23.5 for an example COC form.

### 17.3 Laboratory Required Paperwork

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Approved By:                      **Area Supervisor**



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17.3.1 Data backup logbook and maintenance logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single lineout, which is dated and initialed. Writeovers are NOT acceptable.** Erroneous results must remain legible. A new result is placed near the incorrect result. Blank lines in logbooks must be Z'd out.

## 18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection. In addition to quality control already discussed, the following requirements apply. Corrective action must be taken for unacceptable quality control results based on instructions given in this procedure, and in TriMatrix SOP GR-10-106.

18.2 Dilute and reanalyze all digestates having concentrations greater than the highest standard on the analytical curve.

18.3 For aqueous samples, include a minimum of one digestion blank (BLK) and one blank spike (BS) with each batch of up to twenty samples. For solid samples, include a minimum of one digestion blank (BLK) and one BS with each batch of up to twenty samples. The BLK monitors contamination and carryover while the BS monitors digestion efficiency based on recovery. The BLK and BS must be carried through every step in the digestion and filtration procedure.

18.3.1 A BLK is an aliquot of laboratory reagent water digested as a sample. Any mercury found in the BLK must be at a concentration less than the reporting limit to be acceptable.

18.3.2 A BS is the BLK spiked with a known concentration of mercury.

18.3.2.1 Calculate BS recovery as follows:

$$\text{Percent Recovery} = \frac{C_{\text{Spike}}}{\text{Spike Quantity}} \times 100$$

where:

$C_{\text{spike}}$  = The analyzed concentration of the BS after spiking in ug/L or mg/kg

Spike Quantity = The concentration spiked into the BS in ug/L or mg/kg

18.3.2.2 Recovery of the BS must be within 85 – 115% of the expected value for method reference 245.1 and within 80 – 120% for method reference 7470A and 7471A.

18.4 Matrix spike and matrix spike duplicates are a sample spiked with a known mercury concentration. Prepare and analyze one matrix spike (MS) and one matrix spike duplicate (MSD) with each batch of up to twenty samples. The matrix spike monitors digestion efficiency in the

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 QA Officer

Approved By: DL 4-6-10  
 Area Supervisor



specific sample matrix. A matrix spike duplicate is a replicate of the matrix spike using the same sample and monitors precision within the sample matrix.

18.4.1 Calculate matrix spike recovery as follows:

$$\text{Percent Recovery} = \frac{C_{\text{Spike}} - C_{\text{Orig}}}{\text{Spike Quantity}} \times 100$$

where:

$C_{\text{spike}}$  = The quantitated MS concentration in ug/L or mg/kg  
 $C_{\text{orig}}$  = The unspiked sample concentration in ug/L or mg/kg  
 Spike Quantity = The spiked concentration in ug/L or mg/kg

18.4.2 Calculate precision between two spikes using the following equation:

$$\text{Relative Percent Difference} = \left| \frac{\frac{C_{\text{MSD}} - C_{\text{MS}}}{C_{\text{MSD}} + C_{\text{MS}}}}{2} \right| \times 100$$

where:

$C_{\text{MSD}}$  = The MSD concentration as read from the instrument printout in ug/L or mg/kg  
 $C_{\text{MS}}$  = The MS concentration as read from the instrument printout in ug/L or mg/kg

18.4.3 Matrix spike recovery must be within 80 – 120% of the expected recovery for method reference 7470A/ 7471A and 70 – 130% for method reference 245.1. MSD relative percent difference must be  $\leq 20\%$ .

18.4.4 If MS/MSD criteria fail, reanalyze once. If the second attempt fails, initiate corrective action:

18.4.4.1 Verify the instrument is operating correctly.

18.4.4.2 Verify all other quality control is acceptable.

18.4.4.3 Re-digest the sample and MS/MSD and/or narrate in accordance with TriMatrix SOP GR-10-106.

18.5 Calculate CRL recovery as follows:

$$\text{Percent Recovery} = \frac{C_{\text{Spike}}}{\text{Spike Quantity}} \times 100$$

where:

$C_{\text{spike}}$  = The analyzed CRL concentration in ug/L

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 QA Officer

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 Area Supervisor



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Spike Quantity = The CRL concentration (0.2 ug/L)

## 18.6 The Method of Standard Additions (MSA)

- 18.6.1 Use the method of standard additions for all TCLP extracts having 50% or less matrix spike recovery AND when the leachate concentration is within 20% of the mercury regulatory limit (0.16 – 0.20 mg/L) but not over it.
- 18.6.2 Use the method of standard additions on samples from at least one waste stream of an F-waste delisting petition project. If Section 18.6.1 is not met however, apply the method of standard additions to all samples in the delisting.
- 18.6.3 Standard addition involves adding known mercury concentrations to three sample aliquots. These aliquots are digested and analyzed. The unspiked sample concentration and three spiked aliquots are used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x axis. The absolute value of this point is the sample mercury concentration.
- 18.6.4 Prepare three matrix spikes at 50%, 100%, and 150% of the mercury concentration found in the sample. Final volumes must be the same for each.
- 18.6.5 Digest and analyze to obtain mercury results for each (in ug/L). Using the MSA regression spreadsheet located on the library drive of the facility network, plot absorbance vs. concentration in ug/L. Construct a linear curve without forcing through the origin. The extrapolated x-intercept point is the sample mercury concentration. An example of an MSA plot is shown in Attachment 23.4. For results to be valid, the following limitations must be taken into consideration:
  - 18.6.5.1 The regression curve must be linear, with a correlation coefficient of  $\geq 0.995$  and with a slope less than or equal to 20% of the external calibration curve slope.
  - 18.6.5.2 The chemical form of the mercury added must respond the same way as the mercury in the sample.
  - 18.6.5.3 The interference effect must be constant over the working range of concern.
  - 18.6.5.4 The method of standard additions will not correct for additive interference.

## 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

- 19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

Approved By: <u>          <i>ms</i> 4-2-10          </u>	Approved By: <u>          <i>KE</i> 4-6-10          </u>
QA Officer	Area Supervisor



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SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption	Revision Number: 5.7
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19.1.1 Initial Demonstration of Capability (IDC) Study

- 19.1.1.1 Prepare four second-source blank spikes at a concentration of 2.5 ug/L. Digest, filter and analyze following every step in the procedure.
- 19.1.1.2 Input the four results to the IDC spreadsheet located on the laboratory intranet library.
- 19.1.1.3 Average percent recovery must fall within 85 – 115% recovery for method reference 245.1 and 80 – 120% for method reference 7470A/7471A. Standard deviation of the average must be  $\leq 20\%$  RSD.
- 19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the source of the problem, document changes to the procedure and/or techniques used then repeat the study successfully.
- 19.1.1.5 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed.
- 19.1.1.6 Give a copy of successful IDC spreadsheets and raw data to the quality assurance department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC) Study

- 19.1.2.1 The demonstration of capability study must be repeated annually.
- 19.1.2.2 The CDC may be accomplished in one of the following ways:
  - 19.1.2.2.1 By repeating the IDC study
  - 19.1.2.2.2 By using the last four results from an MDL study performed exclusively by the analyst during the course of routine sample analysis. Only the last four results may be used.
  - 19.1.2.2.3 By using four consecutive blank spike results run during the course of routine sample analysis, exclusively by the analyst.
  - 19.1.2.2.4 By successfully and exclusively analyzing a single-blind performance testing sample during the course of routine sample analysis.

19.2 A method detection limit (MDL) study must be completed annually in accordance with TriMatrix SOP GR-10-125 for each digestion procedure.

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Approved By: <u>          <i>JA</i> 4-2-10          </u>	Approved By: <u>          <i>DA 4-6-10</i>          </u>
QA Officer	Area Supervisor



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SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption	Revision Number: 5.7
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## **20.0 POLLUTION PREVENTION**

- 20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.
- 20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.
- 20.3 Conserve the use of chemicals where applicable.
- 20.4 Comply with all environmental laws associated with chemicals in the laboratory.

## **21.0 WASTE MANAGEMENT**

- 21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.
- 21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.
- 21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

## **22.0 REFERENCES**

- 22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update II, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"*
- 22.2 *Methods for the Determination of Metals in Environmental Samples, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994*
- 22.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update II, Revision I, September 1994, Method 7471A, "Mercury in Solid and Semi Solid Waste (Manual Cold-Vapor Technique)"*

## **23.0 ATTACHMENTS**

- 23.1 Instrument Maintenance Logbook Example
- 23.2 Sample Identification and Weighing Sheet Example – Aqueous
- 23.3 MSA Curve Example
- 23.4 Internal Chain-of-Custody Example

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QA Officer

Approved By:           DCE 4-6-10            
Area Supervisor





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23.5 Sample Identification and Weighing Sheet Example – Solid

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QA Officer	Area Supervisor





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Attachment 23.1  
Instrument Maintenance Logbook Example



PSA Maintenance Logbook

Date	Analyst	Emission/Reference Readings	Changed Pump Tubes	Changed Lamp	Changed Probe	Replaced Sample Valve	Comments
5/6/93	DW						Replaced driver tube
5/16/93	MSS	196.4 104.4	✓				
5/16/93	DW/MSS	200.1 110.4	✓				
5/16/93	MSS	185.1 114.1	✓				
5/16/93	MSS	190.1 112.1	✓				
5/16/93	MSS	195.1 114.1	✓				
5/16/93	MSS	189.4 112.3	✓				
5/16/93	MSS	193.1 113.1	✓				
5/16/93	MSS	193.9 114.3	✓				Replaced GLS line + Driver Tube
5/16/93	MSS	200.1 113.3	✓				
5/16/93	MSS	200.0 112.7	✓				
5/16/93	MSS	190.9 107.9	✓				
5/16/93	MSS	199.5 107.1	✓				
5/16/93	MSS	184.1 110.9	✓				Replaced GLS line + Driver Tube
5/16/93	MSS	190.3 107.9	✓				
5/16/93	MSS	175.1 114.0	✓				
5/16/93	MSS	200.1 115.0	✓				
5/16/93	MSS	200.2 114.8	✓				
5/16/93	MSS	200.5 115.1	✓				

File: MainA\volno.xls

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revision: 1.0

Approved By: DP 4-2-10  
QA Officer

Approved By: DSC 4-6-10  
Area Supervisor





SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption  
EPA Method 245.1, SW-846 Method 7470A, SW-846 Method 7471A  
SOP Number: GR-01-123

Revision Number: 5.7  
Date Revised: 4/2/10  
Date Initiated: 5/6/93

Attachment 23.2  
Sample Identification and Weighing Sheet Example – Aqueous



Metals Laboratory Instrument Run Sequence Hg Aqueous

Instrument #: 216  
Date: \_\_\_\_\_  
Analyst: \_\_\_\_\_  
SnCl #: 8110207

Date File: \_\_\_\_\_  
Calibration BLK/ICB/CCB: 0.5% HNO3 LOT#1108080  
Std/SCV prep date/analyst: \_\_\_\_\_

Standards/SCV prepped daily  
Autosampler Position:

1. BLK/ICB/CCB/8050775
2. STD1/CRL/0.2 ug/L 8050776
3. STD2/0.5 ug/L 8050777
4. STD3/1.0 ug/L 8050778

5. STD4/CCV/2.0 ug/L 8050780
6. STD5/5.0 ug/L 8050781
7. SCV/3.0 ug/L 8100311

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
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32					66				
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34					68				
35					69				
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37					71				
38					72				
39					73				
40					74				
41					75				
42					76				
43					77				
44					78				

Hg\_AQ.xls

revision: 1.0

Approved By: MA 4-2-10  
QA Officer

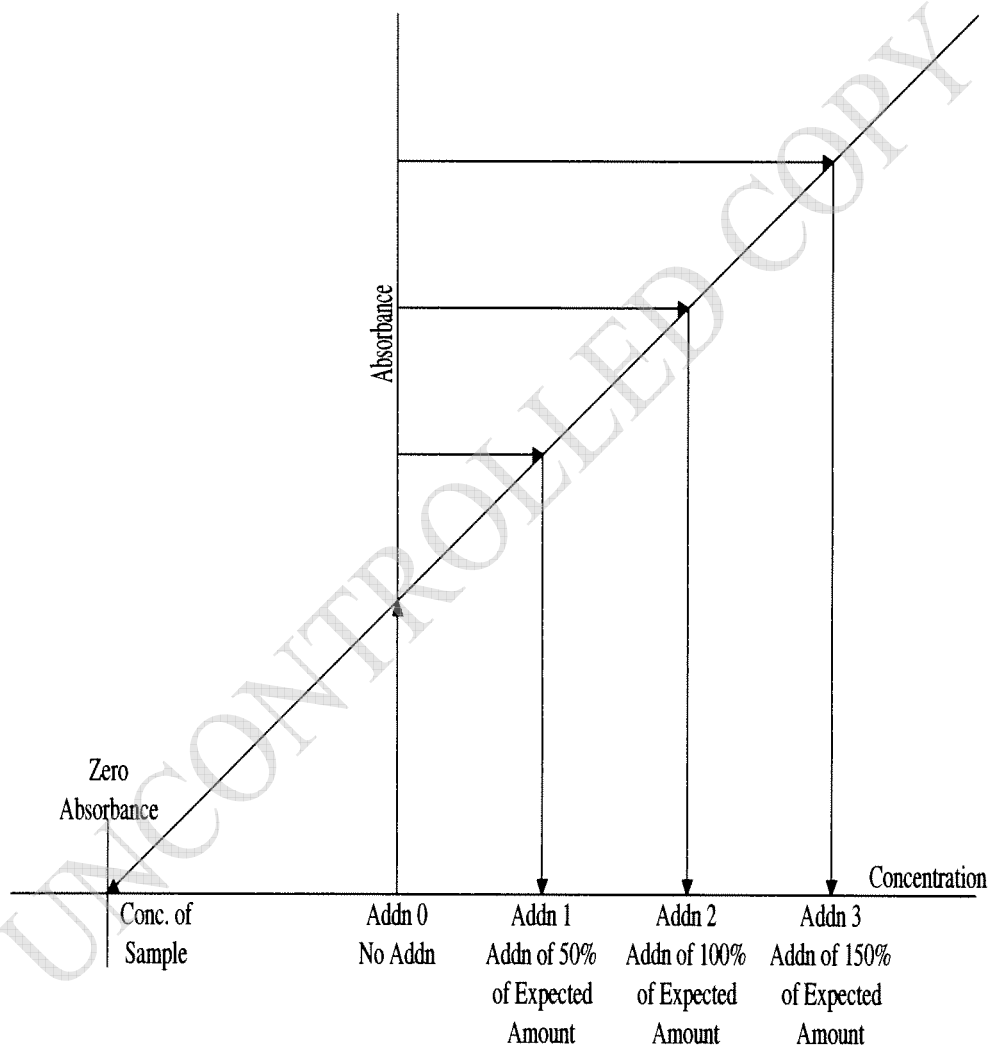
Approved By: DL 4-6-10  
Area Supervisor



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### Attachment 23.3 MSA Curve Example



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 QA Officer

Approved By: DK 4-2-10  
 Area Supervisor





SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption  
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Attachment 23.4  
Internal Chain-of-Custody Example

14-MAY-2003

CLIENT: IDEG-RRD

SUBMITTAL: May 7 & 8, 2003 Soils

Parameter: MERCURY, TOTAL

SAMPLE # REMOVED BY:  
(SIGNATURE)

331322	<u>ma scott</u>
331323	<u>MSS</u>
331324	<u>MSS</u>
331325	<u>MSS</u>
331326	<u>MSS</u>
331327	<u>MSS</u>
331328	<u>MSS</u>
331329	<u>MSS</u>
331330	<u>MSS</u>
331331	<u>MSS</u>
331332	<u>MSS</u>
331333	<u>MSS</u>
331334	<u>MSS</u>
331335	<u>MSS</u>
331336	<u>MSS</u>
331337	<u>MSS</u>

TRIMATRIX LABORATORIES, INC.  
CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

PROJECT: Former Osceola Refinery  
West Branch-Sag. Bay Dist.  
PROJECT: 33923-7

Method: CV/MS/7471/SOL

Ref Cit: USEPA-7471A

DATE & TIME  
REMOVED

RELINQUISHED BY:  
DATE & TIME

Matrix: SOIL

RECEIVED BY:  
DATE & TIME

DATE & TIME  
RETURNED

05-05-03 1:30 PM

05-05-03 3:30 PM

05-13-03 3:30 PM

05-15-03 3:30 PM

Approved By: MA 4-2-10  
QA Officer

Approved By: DEC 4-6-10  
Area Supervisor





SOP Name: Mercury by semi-Automated Cold-Vapor Atomic Absorption  
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Date Initiated: 5/6/93

Attachment 23.5  
Sample Identification and Weighing Sheet Example – Solid



Metals Laboratory Instrument Run Sequence Hg Solids

Instrument #: 216  
Date: \_\_\_\_\_  
Analyst: \_\_\_\_\_  
SnC#: 8110207

Data File: \_\_\_\_\_  
Calibration BLK/ICB/CCB: 0.5% HNO3 LOT#1108080  
Std/SCV prep date/analyst: \_\_\_\_\_

Standards/SCV prepped daily  
Autosampler Position:

1. BLK/ICB/CCB/8050782
2. STD1/CRL/0.2 ug/L 8050784
3. STD2/0.5 ug/L 8050786
4. STD3/1.0 ug/L 8050788

5. STD4/CCV/2.0 ug/L 8050789
6. STD5/5.0 ug/L 8050790
7. SCV/3.0 ug/L 8100310

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
21					55				
22					56				
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40					74				
41					75				
42					76				
43					77				
44					78				

Hg\_solid.xls

revision: 1.0

Approved By: MA 4-2-10  
QA Officer

Approved By: OK 4-6-10  
Area Supervisor





## STANDARD OPERATING PROCEDURE

### Inductively Coupled Plasma-Mass Spectrometry Perkin Elmer Elan 6000/6100

#### SW-846 Method 6020A

#### APPROVALS:

Area Supervisor: Denise S. Coffey Date: 2-22-10  
QA Officer: Tom C. Boocher Date: 2-22-10  
Operations Manager: Jeff P. Glaser Date: 2/23/10

Procedure Number: GR-01-129  
Revision Number: 3.9

Date Initiated: 12/4/97  
Effective Date: 3/10/10

Date Revised: 2/22/10  
Pages Revised: All

By: Denise S. Coffey

Total Number of Pages: 34

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
_____	_____	_____
_____	_____	_____
_____	_____	_____



SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
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## 1.0 SCOPE AND APPLICATION

- 1.1 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) is applicable to a large range of metallic elements in numerous matrices, including but not limited to soil, non-potable water, TCLP extracts, oil, solvents, sludge and air. Most matrices require solubilization or digestion prior to analysis.

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Draft Update IVA, May 1998, Method 6020A, "Inductively Coupled Plasma – Mass Spectrometry", Revision 1, January 1998*

## 3.0 SUMMARY OF PROCEDURE

- 3.1 Prior to analysis, samples are solubilized or digested using appropriate sample preparation. Refer to TriMatrix standard operating procedures for metals sample preparation and digestion.
- 3.2 This procedure describes the multi-elemental determination of analytes by ICP-MS. The procedure measures ions produced by a radio frequency inductively coupled plasma. Sample material in solution is introduced by pneumatic nebulization into radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. Ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of mass-to-charge ratio by a quadrupole. Ion detection is by an electron multiplier or Faraday detector and the information is processed through data handling software.
- 3.3 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or from the sample matrix. Instrumental drift, suppressions or enhancements of instrument response caused by the sample matrix must be corrected by the use of internal standards.

## 4.0 PARAMETER OR COMPOUND LIST

- 4.1 This procedure may be used for analysis of the following metals.

Aluminum	Barium	Cadmium	Copper	Molybdenum	Silver	Vanadium	Strontium
Antimony	Beryllium	Chromium	Lead	Nickel	Thallium	Zinc	Gold
Arsenic	Boron	Cobalt	Manganese	Selenium	Tin	Uranium	Zirconium

## 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

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- 5.2 ELAN 6000 *Software Guide*; Perkin Elmer Corporation, Manual 0993-8965, Release E, June 1995
- 5.3 ELAN 6000 *Hardware Guide*; Perkin Elmer Corporation, Manual 0993-8969, Release D, September 1995
- 5.4 TriMatrix SOP GR-01-137, *Acid Digestion of Sediments, Sludges, Soils and Organic Waste using Block Digestion*, latest revision
- 5.5 TriMatrix SOP GR-01-144, *Block Digestion of Antimony in Sediments, Sludges and Soils*, latest revision
- 5.6 TriMatrix SOP GR-01-147, *Acid Digestion of Total/Dissolved Water TCLP/SPLP Extract and Wastewater by Block Digestion for ICP Total Metals Analysis Acid Digestion of Total/Dissolved Water and TCLP/SPLP Extract By Block Digestion for ICP/MS Total Metals Analysis*, latest revision
- 5.8 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision
- 5.9 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision
- 5.10 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

## 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio ( $m/z$ ). All elements determined by this procedure have at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use, only molybdenum<sup>98</sup> (ruthenium) and selenium<sup>82</sup> (krypton) have isobaric elemental interferences. If alternative isotopes having higher natural abundance are selected to achieve greater sensitivity, an isobaric interference may occur. A data software system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element, and subtracting the appropriate signal from the analyte isotope signal. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for calculations. Relevant isotope ratios must be established prior to the application of any corrections. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, analysis using another verified and documented isotope, or use of another method.
- 6.2 Abundance sensitivity is a property defining the degree to which mass peak wings contribute to adjacent masses. Abundance sensitivity is affected by ion energy and quadropole operating pressure. Wing overlap interferences may result when a small ion peak is measured adjacent to a large one. The potential for these interferences must be recognized and spectrometer resolution adjusted to minimize them.
- 6.3 Isobaric molecular and doubly charged ion interferences in ICP-MS are caused by more than one atom or charge, respectively. Most isobaric interferences that affect ICP-MS determinations have been identified in the literature. Examples include  $\text{ArCl}^+$  ions on the  $^{75}\text{As}$  signal and  $\text{MoO}^+$  ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using natural

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isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<2%) counting statistics. Because the  $^{35}\text{Cl}$  natural abundance of 75.77% is 3.13 times the  $^{37}\text{Cl}$  abundance of 24.23%, the chloride corrections for arsenic can be calculated (approximately) as follows, where the  $^{38}\text{Ar}^{37}\text{Cl}+$  contribution at  $m/z$  is negligible 0.06% of the  $^{40}\text{Ar}^{35}\text{Cl}+$  signal:

Corrected arsenic signal (using natural isotope abundances for coefficient approximations) =  

$$[(m/z\ 75\ \text{signal}) - (3.13)(m/z\ 77\ \text{signal}) + (2.73)(m/z\ 82\ \text{signal})]$$

Where the final term " $(2.73)(m/z\ 82\ \text{signal})$ " adjusts for any selenium contribution at  $m/z\ 77$

- 6.3.1 Cadmium values will be biased low by this type of equation when  $^{97}\text{ZrO}^+$  ions contribute at  $m/z\ 108$ , but  $m/z\ 111$  for cadmium is even subject to direct ( $^{94}\text{ZrO}^+$ ) additive interferences when Zr is present.
- 6.3.2 The coefficients in the cadmium equation are ONLY illustrative for the arsenic equation above. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for the standard solution at a concentration providing suitable (<1%) counting precision.
- 6.3.3 The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable. For example, oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by use of an appropriate oxide as the interferent. This type of correction has been reported for oxide-ion corrections using  $\text{ThO}^+/\text{Th}^+$  for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided reporting limits, accuracy and precision requirements for analysis can be met.
- 6.3.4 If analysis requirements are not met, ionic interference is corrected based on actual instrument response instead of literature equations. Instrument-derived equations may be updated as needed without change to the standard operating procedure.
- 6.3.5 Ionic interference corrections are printed with each quantitative analysis calibration report after the correlation coefficient report. Refer to Attachment 23.11 for an ionic interference correction report example.
- 6.4 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

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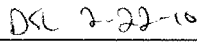
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- 6.5 Memory interferences – Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from material buildup in the plasma torch and spray chamber. The site where these effects occur is element dependent and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interference must be recognized within an analytical run and suitable rinse times used to reduce them. Memory interferences must be monitored within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alert to the possibility of a memory effect and must examine analyte concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample must be reanalyzed after a long rinse period.

## 7.0 SAFETY PRECAUTIONS

- 7.1 Analysts must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.
- 7.2 Concentrated acids are used in the preparation of standards and samples for analysis by ICP-MS. Disposable gloves, laboratory coats and approved safety glasses must be worn at all times when handling concentrated acids. Disposable gloves must also be worn when handling digested samples. Refer to the MSDS for information on these or any other chemical utilized in this procedure.
- 7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, extinguish the plasma if lit and immediately contact the metals laboratory manager. Under no circumstances must the ICP-MS be used if the exhaust hood is not working.
- 7.4 The plasma emits UV radiation. Avoid looking directly at the plasma without some type of strong UV protection. The instrument uses a UV resistant material in the viewing port so the analyst may watch the plasma. Do not tamper with this plate. Do not operate the machine without this plate in place. Do not attempt to view the plasma directly or indirectly in any way. Failure to follow this policy may cause serious and immediate damage to the retina of the eye and will result in disciplinary action.
- 7.5 The ICP-MS emits a strong RF field. To minimize exposure to this field, Perkin Elmer has included several safety interlocks to prevent direct exposure to harmful radiation. Never override any interlock on the ICP-MS. When working on the instrument, replace all RF shielding using all supplied screws. If any safety device has been tampered with, contact the metals lab manager.
- 7.6 The ICP uses argon to sustain plasma and to nebulize sample. Argon can cause suffocation through oxygen replacement. It is therefore imperative that all sources of argon be turned off with a valve when not in use. Since argon is colorless and odorless, if a feeling of lightheadedness develops, evacuate the metals laboratory at once and notify the area supervisor and/or the health and safety officer. Refer to the MSDS for information on this or any chemicals utilized.
- 7.7 All elements used in this procedure are toxic. Refer to the MSDS for information on these or any other chemicals utilized in this procedure.

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7.8 No food or drink is allowed in the metals laboratory.

7.9 Wash hands before starting work. Chemicals may be present on the skin, which may interfere with metals analysis. Wash hands before leaving the metals laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a second party through casual contact.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Aqueous samples should have been acidified at the time of collection to a pH of  $<2$ . If the pH is not  $<2$  when received by the laboratory, the sample pH must be lowered to  $<2$  during the sample receipt process. A minimum of 24 hours must elapse after adjustment prior to sample digestion.

8.2 Solid samples require no preservation for metals. However, if the same sample container is scheduled for other analyses where refrigeration of the sample is needed, store at  $4 \pm 2^{\circ}\text{C}$ .

8.3 Samples may be collected in glass or polyethylene. If Silicon or Boron is to be analyzed, polyethylene containers must be used. Acidified sample must never come into contact with metal, to prevent contaminants leaching into the sample.

8.4 For dissolved metals analysis, the sample should have been filtered on site and acidified to pH  $<2$ . If pH is not  $<2$ , the sample pH must be lowered to  $<2$  at sample receipt and a minimum of 24 hours must elapse after the adjustment, before analyzing the sample.

8.5 Holding times for all metals for ICP-MS is 180 days. In the case of TCLP or similar extracts, the holding time starts at the time leachate is generated.

8.6 The minimum sample volume for this procedure is 50 mL and 150 mL if samples require matrix QC. Samples must be solubilized prior to analysis. A smaller minimum volume is required if a sample will need diluted due to high concentration. Small minimum volumes sacrifice the reporting limit.


8.7 Digested (solubilized) samples and undigested acidified aqueous samples need not be refrigerated. All samples must be at room temperature prior to digestion. All digestates must be at room temperature prior to analysis.

## 9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Perkin Elmer, ELAN 6000/6100 inductively coupled plasma mass spectrometers (ICP-MS):

9.1.1 Both instruments are capable of scanning the mass range 6-240 amu with a minimum resolution of 1 amu peak width at 10% peak height. Both instruments are fitted with an extended dynamic range detection system. The instruments also include an ELAN data system capable of making corrections for isobaric interferences and application of the internal standards technique.

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NOTE: Electron multiplier detectors are used and precautions must be taken to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

- 9.1.2 Radio-frequency generators are used, compliant with FCC regulations.
- 9.1.3 A variable-speed peristaltic pump delivers solution to the nebulizer.
- 9.1.4 A mass-flow controller on the nebulizer gas supply is used.
- 9.2 Analytical balance with the capability of measuring to 0.1 mg for use in weighing solids, for preparing standards and for determining dissolved solids in digestates or extracts.
- 9.3 A Recirculator/chiller (Neslab CFT750). Temperature must be maintained at 18° C and pressure must be 35-60 psi. The recirculator is attached directly to the mass spectrometer to maintain isothermal conditions.
- 9.4 Centrifuge tubes (15 mL, clear) or equivalent. The tube must stand upright within the autosampler rack without tipping or leaning.
- 9.5 Eppendorf micropipettes capable of delivering 5-1000 µL
- 9.6 Flasks, Class A volumetric, various sizes
- 9.7 Pipettes, Class A, glass, various sizes
- 9.8 Balance, top-loading, capable of measurement to two decimal places.

## 10.0 ROUTINE PREVENTIVE MAINTENANCE

- 10.1 All maintenance activities must be recorded in the instrument maintenance logbook. Refer to Attachment 23.1 for an example instrument maintenance logbook.
- 10.2 At the beginning of each shift (every 8 hours) the manifold pump tubing must be replaced.
- 10.3 There are two filters on the ELAN 6000/6100 that must be replaced periodically. The two filters are located on the back of the instrument.
- 10.4 Inspect the waste container on the floor by the instrument every shift. If the jug is full, properly dispose of the liquid.
- 10.5 Visually inspect the torch before lighting the instrument. Do not touch or move the torch. The inspection must be visual only.
  - 10.5.1 Open the vacuum chamber.

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- 10.5.2 Observe the torch. If the torch is broken or cracked, replace it. Refer to Section 10.6 for discussion on how to assemble and disassemble the torch.
- 10.5.3 Listen for obvious leaks in the argon lines going to the torch and nebulizer. If leaks are heard, investigate them and tighten the fittings to stop the leak. If a leak cannot be found or cannot be stopped, contact the metals laboratory manager.
- 10.6 Once every four to eight weeks disassemble the entire torch assembly and inspect all parts for wear. Replace all worn or damaged parts. Record all changes in the instrument maintenance logbook.
  - 10.6.1 Turn off the plasma if it is on by selecting instrument, front panel, and then stop on the plasma box. The pump will stop when the plasma is extinguished.
  - 10.6.2 Turn off the RF Generator by turning off the circuit breaker labeled RF Generator (CB1) on the left side of the instrument.
  - 10.6.3 Loosen the retaining ring and remove the spray chamber assembly.
  - 10.6.4 Open the top left cover of the ELAN. Use a flat blade screwdriver on the release mechanism to release the vacuum chamber interlock lever. Grasp the lever and pull in counter clockwise direction to slide the vacuum chamber and interface away from the torch box.
  - 10.6.5 Remove the gas line connections from the torch by loosening the Swagelock fittings. Slide the fittings and connection tubing up and off the torch.
  - 10.6.6 To remove the torch assembly, rotate the torch mount 1/8 turn counter clockwise and remove the entire assembly through the right side of the torch box.
  - 10.6.7 With one hand grasping the torch and the other holding onto the Lexan adapter, twist and pull back on the adapter to separate the adapter from the torch.
  - 10.6.8 Inspect all O-rings and replace any that are cracked or damaged.
  - 10.6.9 Remove the alumina injector. If it is dirty, wash with soap and water. Dry, then put in the muffle furnace at 700° C for 20 minutes.
  - 10.6.10 If the torch is dirty, immerse in an ultrasonic cleaner containing 1% nitric acid. Rinse with metals laboratory reagent water. Replace any torch that cannot be cleaned or is damaged.
  - 10.6.11 After having been cleaned or replaced, reposition and align the ICP torch as follows:
    - 10.6.11.1 Slide the injector/support adapter to the back of the torch.
    - 10.6.11.2 Place the torch through the hole in the right side of the torch box. Rotate the torch assembly 1/8 turn clockwise until the bayonet mount is fully seated.

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- 10.6.11.3 Reattach the gas lines to the ICP torch. Slide both fittings and connecting tubing onto the torch and tighten the Swagelok fittings several turns.
- 10.6.11.4 To check the torch position and measure for proper alignment, follow Sections 10.6.11.5 through 10.6.11.8.
- 10.6.11.5 Place the torch alignment tool on the torch so the alignment tool flange touches the first turn of the load coil. Loosen the torch locking collar approximately 1 cm and slide the torch until it lines up with the outer edge of the alignment tool.
- 10.6.11.6 Slide the tool out approximately 1/2 inch and using the lever, move the vacuum chamber interlock into the lock position.
- 10.6.11.7 Move the vacuum chamber to the closed position to locate the alignment tool within the load coil.
- 10.6.11.8 Move the chamber to the open position and adjust the vacuum chamber position so the top end of the torch is even with the 5.5 mm cutout depth of the alignment tool.
- 10.6.11.9 If the torch is not properly lined up, loosen the two locking screws of the vacuum chamber interlock. Adjust the vacuum chamber forward or backward until the proper position is achieved.
- 10.6.11.10 Slide the vacuum chamber and interface toward the torch box. Secure the vacuum chamber interlock and close the top cover.
- 10.6.11.11 Turn on the RF Generator by turning on the circuit breaker labeled RF Generator (CB1) on the side of the instrument.
- 10.6.12 Inspect the drain line at the base of the spray chamber. There must be no leaks or cracks in the tubing. If a problem exists, replace the O-ring in the drain cap.
- 10.6.13 Inspect the nebulizer for wear.
  - 10.6.13.1 If present, remove salt deposits with water.
  - 10.6.13.2 If a cross-flow nebulizer is used, check the tips for wear. Replace the tips if excessive wear is noted.
- 10.7 Inspect the print quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate cartridge.

## 11.0 CHEMICALS AND REAGENTS

- 11.1 Acids used in standards preparation and for sample processing must be trace metal grade or better. Redistilled acids may be used if demonstrated to be free of contamination.

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- 11.1.1 Concentrated Nitric acid, trace metal grade (Fisher catalog A509-212 or equivalent)
- 11.1.2 Concentrated Hydrochloric acid, trace metal grade (Fisher catalog A508 or equivalent)

11.2 Laboratory reagent water from the MilliQ water system.

11.3 Argon gas supply: Welding grade or better. This is plumbed from a liquid argon tank located outside of the building. Argon is used as the main plasma gas, the auxiliary plasma gas, the nebulizer carrier gas and the RF coil coolant.

## 12.0 STANDARDS PREPARATION

12.1 All standards must be matrix matched as closely as possible to samples and/or sample digestates. All stock standards expire one year after receipt or on the vendor's expiration date, whichever is earlier.

12.2 Two calibration stock solutions identified as QCA and QCB are purchased at certified concentrations of 100 mg/L containing the following elements.

HP2079-1-1L Solution A (QCA)	Aluminum	Beryllium	Chromium	Lead	Selenium	Thallium
	Arsenic	Boron	Cobalt	Manganese	Silver	Vanadium
	Barium	Cadmium	Copper	Nickel	Strontium	Zinc

HP2079-1-1L Solution B (QCB)	Antimony
	Molybdenum
	Tin

12.3 Second-source stock solutions used as the secondary calibration verification (SCV) are purchased as single-element certified solutions at concentrations of 1000 or 10,000 mg/L from an alternate source/vendor.

12.4 The exception to the above is that uranium, gold and zirconium stock calibration and second-source standards are both purchased as single-element certified solutions.

12.5 All working standards and working standard dilutions for the ICP-MS prepared from stock standard solutions expire 24 hours from the time of preparation.

12.6 Prepare all standards using metals-free laboratory reagent water acidified to 1% with trace-metals grade nitric acid (HNO<sub>3</sub>). The acidified water is prepared by adding 10 mL concentrated HNO<sub>3</sub> to 1000 mL laboratory reagent water.

12.7 Prepare uranium, gold and/or zirconium 100 ug/L standards as follows:

12.7.1 Label one bottle "100 mg/L U, Au and/or Zr stock std" then tare on the analytical balance.

12.7.2 Transfer 50 g of 1% nitric acid to the bottle.

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- 12.7.3 Pipette 1.0 mL of 10000 mg/L U, Au and/or Zr stock standard into the bottle then add 1% nitric acid until the final mass is 100 g.
- 12.8 Prepare intermediate and working standards of QCA and/or QCB with U, Au and/or Zr using 1% nitric acid. Rinse nine 125 mL polyethylene bottles with acidified water.
- 12.8.1 Label one bottle 100 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 100 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.2 Label one bottle 50 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 50 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.3 Label one bottle 20 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 20 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.4 Label one bottle 10 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 10 µL of each 100 mg/L standard (labeled QCA, QCB and U, Au and/or Zr) into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.5 Label one bottle 1.0 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 1000 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.6 Label one bottle 0.2 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 200 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.7 Label one bottle 0.04 ug/L. Tare the bottle on an analytical balance. Pour 50 g of 1% nitric acid into the bottle. Pipette 40 µL of the 100 ug/L standard into the bottle. Add 1% nitric acid until the final mass is 100 g.
- 12.8.8 Label one bottle a blank. Tare the bottle on the analytical balance. Pour 100 g of 1% nitric acid into the bottle.
- 12.8.9 Record all standard preparations in element (refer to Attachment 23.2).
- 12.9 Prepare Interference Check Standard A (IFA) as follows:
- 12.9.1 The IFAs are prepared to contain known concentrations of interfering elements that demonstrate the magnitude of interferences and provide an adequate test of any correction of false positives. Chloride in the IFA provides a means to evaluate software corrections for chloride-related interferences such as  $^{35}\text{Cl}^{16}\text{O}^+$  on  $^{51}\text{V}^+$  and  $^{40}\text{Ar}^{35}\text{Cl}^+$  on  $^{75}\text{As}^+$ . Iron is used to demonstrate adequate resolution of the spectrometer for manganese determinations. Molybdenum serves to indicate oxide effects on cadmium isotopes. Other components are present to evaluate the

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ability of the measurement system to correct for various molecular-ion isobaric interferences. The IFA is used to verify that interference levels are corrected by the data system, within quality control limits.

12.9.2 Stock IFA solution is currently purchased from Inorganic Ventures with the following concentrations:

12.9.2.1 6020ICS-OA contains 10,000 mg/L Cl<sup>-</sup>, 2000 mg/L C, 1000 mg/L Al, Ca, Fe, Mg, P, K, Na, S, 20 mg/L Mo and Ti as interferents.

12.9.3 Working IFA and IFB solutions must be prepared fresh daily.

12.9.3.1 IFA is prepared by pipetting 100 µL of 6020ICS-OA to 9.9 mL of 1% nitric acid.

12.9.3.2 IFB is prepared by pipetting 100 µL of 6020ICS-OA and 20 µL of each SCV stock prepared in section 12.10 to 9.9 mL of 1% nitric acid.

12.10 Prepare an SCV Stock Standard Solution as follows:

12.10.1 Rinse three 125 mL polyethylene bottle with 1% nitric acid and tare on the analytical balance.

12.10.2 Transfer approximately 50 g of 1% nitric acid into the bottles.

12.10.3 Pipette 100 µL of each of the following 10000 mg/L second source stock standards into first bottle:

Aluminum	Barium	Cadmium	Copper	Nickel	Thallium	Zinc
Arsenic	Beryllium	Chromium	Lead	Selenium	Silver	Uranium
Strontium	Boron	Cobalt	Manganese	Vanadium	Gold	Zirconium

12.10.4 Pipette 100 µL of 10000 mg/L second source stock standards Antimony and Molybdenum into second bottle.

12.10.5 Pipette 100 µL of 10000 mg/L second source stock standard Tin into the third bottle.

12.10.6 Dilute all three bottles to 100 g with 1% nitric acid.


12.10.7 Concentration for each element is 10 mg/L.

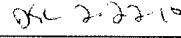
12.11 Prepare the SCV working solution as follows:

12.11.1 Rinse a 125 mL polyethylene bottle with 1% nitric acid.

12.11.2 Tare the bottle on an analytical balance.

12.11.3 Transfer 50 g of 1% nitric acid into the bottle.

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- 12.11.4 Pipette 400  $\mu$ L of each SCV Stock Standard solution into the bottle then add 1% nitric acid until the final mass is 100 g.
- 12.11.5 Refer to Attachment 23.6 for concentrations.
- 12.12 Prepare Internal Standard Solution as follows:
- 12.12.1 For the ELAN 6000, pipette 2000  $\mu$ L Lithium, 400  $\mu$ L Germanium, 40  $\mu$ L Indium, 40  $\mu$ L Terbium, 60  $\mu$ L Bismuth, 60  $\mu$ L Rhodium and 40  $\mu$ L Holmium into 1000 mL of 1% nitric acid.
- 12.12.2 For the ELAN 6100, pipette 2000  $\mu$ L Lithium, 300  $\mu$ L Germanium, 30  $\mu$ L Indium, 20  $\mu$ L Terbium, 40  $\mu$ L Bismuth, 30  $\mu$ L Rhodium and 20  $\mu$ L Holmium into 1000 mL of 1% nitric acid.
- 12.12.3 Internal standard concentrations may be changed at any time, if changing the concentration generates a more appropriate intensity.
- 12.13 Prepare tuning intermediate solution as follows:
- 12.13.1 Rinse a 100 mL polyethylene bottle with 1% nitric acid and tare on the analytical balance.
- 12.13.2 Transfer approximately 50 g 1% nitric acid.
- 12.13.3 Pipette the following standards into the bottle:
- 12.13.3.1 100  $\mu$ L of 10000 mg/L thallium
- 12.13.3.2 100  $\mu$ L of 10000 mg/L lithium
- 12.13.3.3 100  $\mu$ L of 10000 mg/L cobalt
- 12.13.3.4 100  $\mu$ L of 10000 mg/L barium
- 12.13.3.5 100  $\mu$ L of 10000 mg/L cerium
- 12.13.4 Add 1% nitric acid to a final mass of 100 g. Cap the bottle and shake.
- 12.13.5 Rinse a 1000 mL polyethylene bottle with 1% nitric acid and tare on the top-loading balance.
- 12.13.6 Transfer approximately 500 g 1% nitric acid.
- 12.13.7 Pipette the following standards into the bottle:
- 12.13.7.1 10  $\mu$ L of 1000 mg/L indium
- 12.13.7.2 1000  $\mu$ L of 6020A intermediate solution

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
12.13.8 Add 1% nitric acid to a final mass of 1000 g. Cap the bottle and shake.

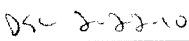
### 13.0 SAMPLE PREPARATION

- 13.1 Soil, sludge and waste samples must be digested in accordance with TriMatrix SOP GR-01-137 and GR-01-144.
- 13.2 Aqueous samples that need digested such as groundwater, TCLP and SPLP leachates must be digested in accordance with TriMatrix SOP GR-01-147.
- 13.3 Air samples are digested as specified by the method used in sample collection.
- 13.4 Any dissolved metals sample having an odor, precipitate or turbidity greater than 1 NTU after filtering must be digested prior to analysis.

### 14.0 CALIBRATION PROCEDURES

- 14.1 The ELAN 6000 and 6100 must be calibrated for every analytical run or every 24 hours, whichever is more frequent.
- 14.1.1 Initial calibration consists of an 8-point calibration curve for silver and cadmium. A 7-point curve is used for all other elements. An initial calibration curve consists of the standard series prepared in Section 12.4. Silver and cadmium curves include the 0.04 ug/L standard. This concentration is not included in the calibration for other elements. The 0 ug/L blank is included as a standard in all calibrations.
- 14.1.2 The average of at least three plasma responses for each concentration is used in generating calibration curves.
- 14.1.3 In all cases, calibration is performed using a multi-point linear regression. The correlation coefficient (r) must be greater than or equal to 0.995. If the coefficient is less than 0.995, recalibrate or postpone analysis (for only the out-of-control elements) until an acceptable calibration can be attained.
- 14.1.4 The highest concentration in the calibration range must not exceed 90% of the linear range (refer to Section 18.10).
- 14.2 After calibration, an Initial Calibration Verification is performed.
- 14.2.1 Analyze the Second-Source Calibration Verification (SCV). The SCV must be prepared from an independent (second source) material to a concentration near the mid-point of the calibration curve. The SCV control window is 90%-110% of the expected value. If the percent recovery of any target element does not fall within the control window, re-analyze one time. If the re-analysis does not pass recalibrate the instrument and restart analysis at Section 14.2 or postpone analysis (for only the out-of-control elements) until an acceptable calibration can be obtained.

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
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Area Supervisor




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- 14.2.2 Analyze the Initial Calibration Blank (CCB). A CCB is quantitation of the 0 ug/L blank (prepared in Section 12.4.9), as a sample. The absolute value of the CCB reading must be less than 1/5<sup>th</sup> the minimum reporting limit required for analysis. If the CCB is not lower than 1/5<sup>th</sup> the reporting limit, re-analyze one time. If the re-analysis does not pass recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated. If the ICB remains out-of-control, see the metals laboratory supervisor.
- 14.2.3 Analyze the MRL-level Calibration Verification (CRL). A CRL is a reporting level standard with a concentration at the minimum reporting limit (MRL). The control window is 50%-150% of the expected value. If percent recovery falls outside the 50-150% window, a narrative must be attached to the analytical batch.
- 14.2.4 Analyze the IFA solution which is comprised of interfering elements only. The IFA demonstrates the magnitude of interferences and provides a test of any corrections made. Control limits for the IFA are  $\pm 3$  times the reporting limit for non-spiked elements.
- 14.2.5 Analyze Interference Check Standard B (IFB). The IFB contains a known concentration of interfering elements that monitors analyte recovery and/or detection, and provides a test for any corrections that need done.
- 14.2.5.1 Control limits are 90-110% of the known concentration and  $\pm 3$  times the reporting limit for non-spiked elements.
- 14.2.5.2 If the IFB is outside a control limit, stop the analysis and remake the solution then re-analyze successfully to continue with sample analysis.
- 14.2.5.3 If the IFB fails again, generate an acceptable calibration and repeat the analysis for all out-of-control elements. Alternatively, postpone analysis until an acceptable calibration can be generated.
- 14.2.6 Following the IFB, analyze a Continuing Calibration Verification (CCV) standard. A CCV is the same solution as the SCV. The control window is 90%-110% of the expected value. If the CCV is not within the control window, stop the analysis and re-analyze one time. If the CCV re-analysis is within the control window, continue the analysis. If the CCV remains out-of-control, recalibrate the instrument for the affected elements and restart the analysis at Section 14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated.
- 14.2.7 Follow the CCV with analysis of a Continuing Calibration Blank (CCB). A CCB is quantitation of the 0 ug/L blank prepared in Section 12.4.9, as a sample. The absolute value of the CCB reading must be less than 1/5<sup>th</sup> the lowest reporting limit required for analysis. If the CCB is not lower than 1/5<sup>th</sup> the reporting limit, stop the analysis and re-analyze one time. If the CCB re-analysis is less than 1/5<sup>th</sup> the reporting limit, continue sample analysis. If the CCB remains out-of-control, recalibrate the instrument for the affected elements and restart the analysis at Section

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14.2 or postpone analysis (for only out-of-control elements) until an acceptable calibration can be generated.

- 14.2.8 Analyze up to ten samples. If percent RSD for any result obtained from the replicate plasma response is >20, the sample run immediately prior to the result must be checked for high analyte concentration. If the analyte is high, check the triple-replicate result in question to verify that concentration is not decreasing. If concentration is decreasing, reanalyze the sample. If the concentration is not decreasing, report the result in question.
- 14.2.9 After each analysis of up to ten samples, analyze the CCV. If the CCV is not within the control window, stop the analysis and re-analyze one time. If CCV re-analysis is within the control window, continue sample analysis. If CCV re-analysis is out-of-control, recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements), until an acceptable calibration can be generated. All samples associated with an unacceptable CCV must be re-analyzed for all affected target elements.
- 14.2.10 After each CCV, analyze the CCB. If a CCB is not less than 1/5<sup>th</sup> the reporting limit, stop the analysis and re-analyze one time. If CCB re-analysis is within the control window, continue sample analysis. If CCB re-analysis is out-of-control, recalibrate the instrument and start again at Section 14.2 or postpone analysis (for only out-of-control elements), until an acceptable calibration can be generated. All samples associated with an unacceptable CCB must be re-analyzed for all affected target elements.
- 14.2.11 Repeat Sections 14.2.8 through 14.2.10 until all samples have been run. The analytical run must be concluded with analysis of acceptable CCV and CCB results.
- 14.2.12 The IFA and IFB solutions must be re-verified if an analytical run exceeds 12 hours.

## 15.0 ANALYTICAL PROCEDURE

- 15.1 The following sections assume the user to be familiar with ELAN 6000/6100 operating software. If unfamiliar or unsure of any particular part of the operation, consult the instrument manual for additional instructions. This section provides adequate instruction for normal instrumental operation but is not intended to replace the instrument operating manual.
- 15.2 Preliminary treatment of all sample matrices is almost always necessary to solubilize analyte.
  - 15.2.1 Digestions are referenced in Section 5.0.
  - 15.2.2 A minimum of five internal standards must always be used.
- 15.3 Once the plasma has been on for at least 30 minutes, daily tuning of the instrument needs performed. However, if the sample and skimmer cones have been changed, a more rigorous tune needs performed as follows:
  - 15.3.1 Realign the x and y cone axis.

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- 15.3.2 Optimize the nebulizer flow and lens voltage.
- 15.3.3 Tune the mass spec over the entire mass range.
- 15.3.4 Check instrument performance:
  - 15.3.4.1 The instrument will take six readings to generate a percent relative standard deviation (RSD) value for lithium, cobalt, indium and thallium. For each element, RSD must be less than 5.0. If not, repeat the evaluation once. If any element is greater than or equal to 5.0, locate and correct the problem before proceeding.
  - 15.3.4.2 Calculate Percent  $Ba^{2+}/Ba^0$  and  $CeO/Ce^0$ . If either criterion is greater than or equal to 3.0%, repeat the check once. If one or the other remains out of limits, locate and correct the problem then repeat the tune.
- 15.4 Once all tune optimization steps are complete, calibrate in accordance with Section 14.0 and run samples.

## 16.0 CALCULATIONS AND DATA HANDLING

- 16.1 Collect data from the instrument and transfer to the laboratory information management system in accordance with TriMatrix SOP GR-10-123.
- 16.3 Whenever possible, calculate a result from both primary and secondary isotopes. Report only the primary isotope result. However, if there is greater than 25% difference between the two values, report the lowest value as the result. Monitor percent difference as follows:

$$\frac{\text{primary ion} - \text{secondary ion}}{\text{primary ion}} \times 100 = \text{percent difference}$$

## 17.0 DATA REPORTING AND DELIVERABLES

- 17.1 Analysts running samples are responsible for correctly filling in, handing in and filing all associated paperwork. It is essential to perform these tasks conscientiously to provide clients with defensible data.
- 17.2 Report data to the laboratory information management system (Element™) in accordance with TriMatrix SOP GR-10-123.
  - 17.2.1 Data must be recorded completely to ensure that results are reported correctly and data is associated with the right quality control batch. It is important that all required information be recorded correctly and completely.
  - 17.2.2 Quality control information must be completed and recorded including spiked concentrations where applicable.

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- 17.2.3 If internal chain-of-custody (CoC) is required, it is important that CoC forms be filled in correctly and archived appropriately. Refer to Attachment 23.5 for an internal CoC form example.
- 17.2.4 All analysis hardcopy and benchsheets must be archived appropriately.
- 17.3 Laboratory Required Paperwork
- 17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a write-over, and must be dated and initialed. Blank lines in logbooks must be Z'd out.
- 18.0 QUALITY ASSURANCE**
- 18.1 All quality control data must be maintained and available for easy reference or inspection.
- 18.2 Instrument detection limit (IDL) studies must be performed quarterly.
- 18.2.1 Analyze seven sequential aliquots of metals reagent water. Repeat on two more non-consecutive days to obtain 21 results in 3 datasets.
- 18.2.2 Input these data to the IDL spreadsheet located on the laboratory intranet to calculate the IDL which is the calculated average standard deviation.
- 18.2.3 The spreadsheet compares the calculated IDL to the calculated MDL. The IDL must be less than the MDL. If not, locate the source of the problem and repeat the IDL/MDL studies successfully. Repeat only for elements that failed.
- 18.2.4 Consult the metals supervisor and/or the quality assurance department if repeated studies indicate the IDL is not less than the MDL.
- 18.3 Dilute and re-analyze samples that are more concentrated than the highest standard on the calibration curve.
- 18.4 Include a minimum of one Blank (BLK) per sample batch digested to determine if contamination or any memory effects are occurring. The BLK must be carried through all sample preparation steps. Results must be below the reporting limit.
- 18.5 Include a minimum of one Laboratory Control Sample (BS) per sample batch. The BS must be carried through all sample preparation steps. A BS is reagent water spiked with each element between the low and mid-level calibration range. Percent recovery is not to exceed 80%-120% recovery of the expected value. If a BS is outside the control window, it must be re-run one time. If the re-analysis is outside the control window all samples associated with the BS must be re-prepared and re-analyzed.
- 18.6 Analyze one matrix spike (MS) and one matrix spike duplicate (MSD) at a frequency of at least 1 in 20 (5%). MS/MSDs are sample aliquots to which is added a known analyte concentration between the low and mid-level calibration range. Calculate percent recovery for each element. Percent recovery is not to exceed

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75%-125% recovery with an RPD <20%. Matrix spiking monitors sample preparation efficiency relative to each matrix type. Relative percent difference (RPD) is calculated from the MS/MSD duplication. The RPD value monitors precision within sample preparation and analysis.

18.6.1 Pretreated samples must be spiked before digestion begins.

18.6.2 Samples not requiring pretreatment are spiked before loading on the autosampler tray.

18.6.2.1 Two "spiking solutions" are used to perform matrix spikes. These solutions are stable but due to high analyte concentrations are not compatible when mixed.

18.6.3 Calculate MS/MSD percent recovery as follows:

$$\%R = \frac{C_{\text{Spike}} - C_{\text{Orig}}}{\text{Spike Concentration}} \times 100$$

where:

%R = Spike recovery, in %  
 $C_{\text{Spike}}$  = Concentration of the spiked sample  
 $C_{\text{Orig}}$  = Concentration of the unspiked sample  
 Spike Concentration = Concentration of the element spiked into the sample

18.6.4 Calculate MS/MSD relative percent difference as follows,

$$\%RPD = \frac{C_{\text{MSD}} - C_{\text{MS}}}{(C_{\text{MSD}} + C_{\text{MS}})/2} \times 100$$

where:

%RPD = Relative percent difference between the MS and MSD  
 $C_{\text{MSD}}$  = Concentration of the MSD as read from the instrument data printout  
 $C_{\text{MS}}$  = Concentration of the MSD as read from the instrument data printout

18.6.5 A matrix spike must recover within 75-125% of the expected value. Precision between two matrix spikes must be ≤20%. Refer to TriMatrix SOP GR-10-106 for corrective actions.

18.7 The following tests must be performed with each sample batch to ensure that neither positive nor negative interferences are affecting element measurement or distorting reported value accuracies.

18.7.1 Post Spike (PS): The same sample from which the MS/MSD aliquots were prepared must also be spiked with a Post Spike. An analyte spike between the low and mid-range of the calibration curve is added to a portion of prepared digestate or digestate dilution. The control window is 80%-120% of the expected value. If MS/MSD recovery is acceptable and the PS is unacceptable, a serial dilution must be performed on the sample. If MS/MSD recovery is unacceptable and PS recovery is unacceptable then a matrix effect is confirmed and the sample qualified accordingly.

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- 18.7.2 Serial Dilution: A serial dilution must be performed on the same sample as the MS/MSD, if analyte concentration is  $\geq 100$  times the IDL. Analysis of a 1:5 dilution must agree within  $\pm 10\%$  of the original determination. If not, a chemical or physical interference effect must be suspected and the sample qualified accordingly.
- 18.7.3 The method of standard additions involves adding known concentrations of a standard to three digestate aliquots. The three results are then used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x-axis. The value of this point is the analyte concentration in the sample.
- 18.7.4 To perform the method of standard additions, prepare three spikes at 50%, 100% and 150% of the concentration found in the processed sample.
- 18.7.5 Analyze the spikes and the non-spiked processed sample. Using the regression program located on the laboratory intranet library, plot absorbance against concentration to construct a regression curve. The extrapolated x-intercept of the regression curve is the analyte concentration in the sample. Refer to Attachment 23.9 for a plot example. For results to be valid, the following limitations must be taken into consideration:
- 18.7.5.1 The curve constructed must be linear, with a correlation coefficient of  $\geq 0.995$ .
- 18.7.5.2 The chemical form of the analyte added must respond the same way as the analyte in the sample.
- 18.7.5.3 The interference effect must be constant over the working range of concern.
- 18.7.5.4 The signal must be corrected for any additive interference.
- 18.7.5.5 The method of standard additions must be performed on any TCLP sample whose spike recovery is less than 50% and with a concentration less than or equal to the regulatory limit. The method of standard additions is also required when a TCLP extract concentration is within 20% of the regulatory limit. TCLP regulatory limits are as follows:

Element	Regulatory Limit (mg/L)	Element	Regulatory Limit (mg/L)
Arsenic	5.0 mg/L	Lead	5.0 mg/L
Barium	100 mg/L	Selenium	1.0 mg/L
Cadmium	1.0 mg/L	Silver	8.0 mg/L
Chromium	5.0 mg/L	Zinc	500 mg/L
Copper	100 mg/L		

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*SW 2-22-10*  
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- 18.7.5.6 The method of standard additions does not detect coincidental spectral overlap. If overlap is suspected, use computer compensation, an alternative wavelength or comparison with an alternate method to determine and/or correct.
- 18.7.6 Duplicate Analysis (DUP): If specified for a project, a DUP is analyzed as a substitute for, or in addition to the MSD. A DUP is a second sample aliquot analyzed in the same way as the first. The acceptance limit for DUP RPD is  $\leq 20\%$ .
- 18.8 Verify instrument calibration by analyzing appropriate quality control verification standards as described in Section 14.0.
- 18.9 Internal standardization (IS) must be used in all analyses to correct for instrument drift and physical interferences. A minimum of five internal standards are used at all times. Recovery for each internal standard is 70-120% of the expected value.
- 18.9.1 If an internal standard has low recovery ( $< 70\%$ ), do a 1:5 dilution of the sample or digestate and re-analyze. If internal standard recovery is below 70%, do another 1:5 dilution. Repeat until acceptable internal standard recovery is achieved.
- 18.9.2 If an internal standard recovery is high ( $> 120\%$ ), the element may be present in the sample. Use an alternate internal standard.
- 18.9.3 All internal standards must be within 50 amu of the element of interest.
- 18.10 Linear range studies must be performed semi-annually or more frequently if specified by any project DQO, or when there is a significant change in instrument response.
- 18.10.1 To perform a linear range study, calibrate the instrument as for sample analysis.
- 18.10.2 Run successive standards at an increasingly higher concentration and calculate percent recovery for each element. Begin by running a 200 ug/L standard.
- 18.10.3 If percent recovery is acceptable (90-110%) for the 200 ug/L standard, run a higher concentration. Continue until percent recovery is not acceptable then run a standard at a lower concentration but higher than the last acceptable standard to zero in on the maximum value in the linear range.
- 18.10.4 The highest concentration run with acceptable percent recovery is the linear range.

## 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

- 19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

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Approved By: <u>          <i>M</i> 2-22-10          </u>	Approved By: <u>          <i>042-2210</i>          </u>
QA Officer	Area Supervisor



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SOP Name: Inductively Coupled Plasma - Mass Spectrometry Perkin Elmer Elan 6000/6100 SW-846 Method 6020A SOP Number: <b>GR-01-129</b>	Revision Number: 3.9  Date Revised: 2/22/10 Date Initiated: 12/4/97
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#### 19.1.1 Initial Demonstration of Capability

- 19.1.1.1 Spike four aliquots of metals reagent water with a second source standard so concentration is in the lower half of the calibration range.
- 19.1.1.2 Digest and analyze the four spikes as samples following every step in the procedures.
- 19.1.1.3 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must be within blank spike control limits. Relative standard deviation must be  $\leq 20\%$ .
- 19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.
- 19.1.1.5 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques uses then repeat the study successfully.
- 19.1.1.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed.

#### 19.1.2 A Continuing Demonstration of Capability (CDC) study must be performed annually by one of the following:

- 19.1.2.1 By repeating the IDC study.
- 19.1.2.2 By using four consecutively analyzed blank spikes (if a second source is used) obtained from routine sample analysis if run exclusively by the analyst.
- 19.1.2.3 By successfully analyzing a performance testing sample during the course of routine sample analysis if run exclusively by the analyst.
- 19.1.2.4 By using the last four of seven results from an MDL study if run exclusively by the analyst. ONLY the last four results may be used.

#### 19.1.3 Submit copies of all successful demonstrations of capability spreadsheets to the quality assurance department for data and training documentation.

#### 19.2 A Method Detection Limit Study must be performed annually in accordance with TriMatrix SOP GR-10-125.

### 20.0 POLLUTION PREVENTION

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Approved By: <u>ms 2-22-10</u>	Approved By: <u>DSC 2-22-10</u>
QA Officer	Area Supervisor



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SOP Name: Inductively Coupled Plasma - Mass Spectrometry Perkin Elmer Elan 6000/6100 SW-846 Method 6020A	Revision Number: 3.9
SOP Number: <b>GR-01-129</b>	Date Revised: 2/22/10 Date Initiated: 12/4/97

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- 20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.
- 20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.
- 20.3 Conserve the use of chemicals.
- 20.4 Comply with all environmental laws associated with chemicals in the laboratory.

## **21.0 WASTE MANAGEMENT**

- 21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located in the library on the laboratory intranet.
- 21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.
- 21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

## **22.0 REFERENCES**

- 22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Draft Update IVA, May 1998, Method 6020A, "Inductively Coupled Plasma – Mass Spectrometry", Revision 1, January 1998*

## **23.0 ATTACHMENTS**

- 23.1 Instrument Maintenance Logbook Example
- 23.2 Standards Log Example
- 23.3 Preparation Batch Report Example
- 23.4 Data Review Report Example
- 23.5 Internal Chain-of-Custody Example
- 23.6 SCV Standard Solution
- 23.7 Element Conditions
- 23.8 Spiking Solution Final Concentration
- 23.9 MSA Curve Example

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Approved By:           *QS* 2-22-10            
QA Officer

Approved By:           *OC 2-22-10*            
Area Supervisor



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SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: **GR-01-129**

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Revision Number: 3.9

Date Revised: 2/22/10

Date Initiated: 12/4/97

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23.10 Ionic Interference Correction Report Example

UNCONTROLLED COPY

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Approved By: ms 2-22-10  
QA Officer

Approved By: da 2-22-10  
Area Supervisor









SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: GR-01-129

Revision Number: 3.9

Date Revised: 2/22/10

Date Initiated: 12/4/97

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### Attachment 23.2 Standards Log Example

#### Analytical Standard Record TriMatrix Laboratories, Inc. A602735

Description:	QCA ICP/MS	Expires:	Mar-31-97
Standard Type:	Analyte Spike	Prepared:	Mar-31-95
Solvent:	Solvent Lot #605319	Prepared By:	** Vendor **
Pipet Volume (mL):	1000	Department:	Expired
Vials:	1	Last Edit:	Jan-30-07 13:48 by DSC
Environmental Response Corp HP2078-1-1L Solution A			
Lot: 605319			
Received 2/28/06			
Analyte	CAN Number	Concentration	Units
Aluminum	7439-00-5	100	mg/L
Arsenic	7440-38-2	100	mg/L
Arsenic, OWEP-SAR	7440-38-2	100	mg/L
Arsenic, OWEP-TAR	7440-38-2	100	mg/L
Barium	7440-39-3	100	mg/L
Beryllium	7440-41-7	100	mg/L
Boron	7440-42-8	100	mg/L
Cadmium	7440-43-9	100	mg/L
Calcium	7440-70-2	1000	mg/L
Chromium	7440-47-3	100	mg/L
Cobalt	7440-48-4	100	mg/L
Copper	7440-50-8	100	mg/L
Iron	7439-89-6	100	mg/L
Lead	7439-92-1	100	mg/L
Lead, Total (Coarse)	7439-92-1	100	mg/L
Lead, Total (Fine)	7439-92-1	100	mg/L
Lithium	7439-93-2	100	mg/L
Magnesium	7439-95-4	1000	mg/L
Manganese	7439-96-5	100	mg/L
Nickel	7440-02-0	100	mg/L
Phosphorus	7723-14-0	100	mg/L
Potassium	7440-09-7	1000	mg/L
Selenium	7782-49-2	100	mg/L
Selenium, OWEP-SAR	7782-49-2	100	mg/L
Selenium, OWEP-TAR	7782-49-2	100	mg/L
Silver	7440-22-3	100	mg/L
Sodium	7440-23-5	1000	mg/L
Strontium	7440-74-6	100	mg/L
Thallium	7440-28-0	100	mg/L
Thallium, OWEP-SAR	7440-28-0	100	mg/L

Reviewed By: \_\_\_\_\_ Date: \_\_\_\_\_

Page 1 of 2

#### Analytical Standard Record TriMatrix Laboratories, Inc. A602735

Thallium, OWEP-TAR	7440-28-0	100	mg/L
Vanadium	7440-62-2	100	mg/L
Zinc	7440-66-6	100	mg/L

Reviewed By: \_\_\_\_\_ Date: \_\_\_\_\_

Page 2 of 2

Approved By: ms 2-22-10  
QA Officer

Approved By: OK 2-22-10  
Area Supervisor





SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: GR-01-129

Revision Number: 3.9  
Date Revised: 2/22/10  
Date Initiated: 12/4/97

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### Attachment 23.3 Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0609650 Page 1 of 2

Printed: 10/3/2007 3:04:45PM

Metals, Water, 3020A Digestion  
(No Surrogate)

Batch Comments: 08/23/06 6:15am-2:30pm

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0608312	Se Total 6020	0608312	Pb Total 6020	0608312	Cr Total 6020
0608312	Cd Total 6020	0608312	As Total 6020	0608312	Ag Total 6020
0608355	Zn Total 6020	0608355	Se Total 6020	0608355	Pb Total 6020
0608355	Cu Total 6020	0608355	Cr Total 6020	0608355	Cd Total 6020
0608355	Ba Total 6020	0608355	As Total 6020	0608355	Ag Total 6020

Lab Number	Comment	Prepared	By	Initial (mL)	Final (mL)	mL Surrogate	Source ID	Spike ID	mL Spike	Client - QC Type	Comments/Comments
0609650-BLK1		Aug-23-06 08:15	LMA	25	125					BLANK	
0609650-B51		Aug-23-06 08:15	LMA	25	125			A602735	25	LC6	
0609650-M61		Aug-23-06 08:15	LMA	25	125		0609650-22	A602735	25	MATRIX SPIKE	
0609650-M6D1		Aug-23-06 08:15	LMA	25	125		0609650-22	A602735	25	MATRIX SPIKE DUP	
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608312-23	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-21	A	Aug-23-06 08:15	LMA	25	125						
0608355-22	C	Aug-23-06 08:15	LMA	25	125						Added for Sequence#23 in 6062623
0608355-22	C	Aug-23-06 08:15	LMA	25	125						
0608355-22	C	Aug-23-06 08:15	LMA	25	125						
0608355-22	C	Aug-23-06 08:15	LMA	25	125						

Comments	Analyst Initials
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TriMatrix Laboratories, Inc.

PREPARATION BATCH 0609650 Page 2 of 2

Printed: 10/3/2007 3:04:45PM

Metals, Water, 3020A Digestion  
(No Surrogate)

Batch Comments: 08/23/06 6:15am-2:30pm

Lab Number	Comment	Prepared	By	Initial (mL)	Final (mL)	mL Surrogate	Source ID	Spike ID	mL Spike	Client - QC Type	Comments/Comments
0609650-22	C	Aug-23-06 08:15		25	125						Added for Sequence#23 in 6062623
0609650-22	C	Aug-23-06 08:15	LMA	25	125						
0609650-22	C	Aug-23-06 08:15	LMA	25	125						
0609650-22	C	Aug-23-06 08:15	LMA	25	125						Added for Sequence#23 in 6062623
0609650-22	C	Aug-23-06 08:15	LMA	25	125						
0609650-F011		Aug-23-06 13:15	DSC	2	10		0609650-22	A603106	50	POST SPIKE	[Spike] 25mL > 125mL, 10mL > 10mL, Spk
0609650-F012		Aug-23-06 14:00	DSC	2	10		0609650-22	A603106	100	POST SPIKE	[Spike] 25mL > 125mL, 10mL > 10mL, Spk

Comments	Analyst Initials
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Approved By: 2-22-10  
QA Officer

Approved By: 2-22-10  
Area Supervisor





SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: GR-01-129

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Revision Number: 3.9

Date Revised: 2/22/10  
Date Initiated: 12/4/97

### Attachment 23.4 Data Review Report Example

TriMatrix Laboratories, Inc.


Data Review Report -- Metals Lab  
Sequence = 6090602 Page 1 of 4

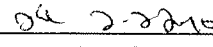
SampleID	Analyte	IRResult	Qualifier	Units	Diln	EResult	EUnits	IMDL	IMDL	EM
6090602-CAL1	Ag Total 6020	0		ug/L	1	0.00	ug/L	0.024	0.04	
6090602-CAL1	Cu Total 6020	0		ug/L	1	0.00	ug/L	0.065	0.2	
6090602-CAL1	Pb Total 6020	0		ug/L	1	0.00	ug/L	0.065	0.2	
6090602-CAL1	Ti Total 6020	0		ug/L	1	0.00	ug/L	0.034	0.2	
6090602-CAL1	V Total 6020	0		ug/L	1	0.00	ug/L	0.17	0.2	
6090602-CAL2	Ag Total 6020	0.04		ug/L	1	0.0400	ug/L	0.024	0.04	
6090602-CAL3	Ag Total 6020	0.200592		ug/L	1	0.201	ug/L	0.024	0.04	
6090602-CAL3	Cu Total 6020	0.2		ug/L	1	0.200	ug/L	0.065	0.2	
6090602-CAL3	Pb Total 6020	0.2		ug/L	1	0.200	ug/L	0.065	0.2	
6090602-CAL3	Ti Total 6020	0.2		ug/L	1	0.200	ug/L	0.034	0.2	
6090602-CAL3	V Total 6020	0.2		ug/L	1	0.200	ug/L	0.17	0.2	
6090602-CAL4	Ag Total 6020	1.000261		ug/L	1	1.00	ug/L	0.024	0.04	
6090602-CAL4	Cu Total 6020	1.002342		ug/L	1	1.00	ug/L	0.065	0.2	
6090602-CAL4	Pb Total 6020	1.00034		ug/L	1	1.00	ug/L	0.065	0.2	
6090602-CAL4	Ti Total 6020	1.000458		ug/L	1	1.00	ug/L	0.034	0.2	
6090602-CAL4	V Total 6020	0.99996		ug/L	1	1.00	ug/L	0.17	0.2	
6090602-CAL5	Ag Total 6020	9.985816		ug/L	1	9.99	ug/L	0.024	0.04	
6090602-CAL5	Cu Total 6020	9.967423		ug/L	1	9.99	ug/L	0.065	0.2	
6090602-CAL5	Pb Total 6020	9.969133		ug/L	1	9.99	ug/L	0.065	0.2	
6090602-CAL5	Ti Total 6020	9.967984		ug/L	1	9.99	ug/L	0.034	0.2	
6090602-CAL5	V Total 6020	9.989488		ug/L	1	9.99	ug/L	0.17	0.2	
6090602-CAL6	Ag Total 6020	20.23713		ug/L	1	20.2	ug/L	0.024	0.04	
6090602-CAL6	Cu Total 6020	20.21078		ug/L	1	20.2	ug/L	0.065	0.2	
6090602-CAL6	Pb Total 6020	20.06969		ug/L	1	20.1	ug/L	0.065	0.2	
6090602-CAL6	Ti Total 6020	20.14292		ug/L	1	20.1	ug/L	0.034	0.2	
6090602-CAL6	V Total 6020	20.08358		ug/L	1	20.1	ug/L	0.17	0.2	
6090602-CAL7	Ag Total 6020	49.5229		ug/L	1	49.5	ug/L	0.024	0.04	
6090602-CAL7	Cu Total 6020	49.47732		ug/L	1	49.5	ug/L	0.065	0.2	
6090602-CAL7	Pb Total 6020	49.94673		ug/L	1	49.9	ug/L	0.065	0.2	
6090602-CAL7	Ti Total 6020	49.54875		ug/L	1	49.5	ug/L	0.034	0.2	
6090602-CAL7	V Total 6020	49.50058		ug/L	1	49.6	ug/L	0.17	0.2	
6090602-CAL8	Pb Total 6020	101.319		ug/L	1	101	ug/L	0.065	0.2	
6090602-ICV1	Ag Total 6020	42.35505		ug/L	1	42.4	ug/L	0.024	0.04	
6090602-ICV1	Cu Total 6020	42.45288		ug/L	1	42.5	ug/L	0.065	0.2	
6090602-ICV1	Pb Total 6020	41.42369		ug/L	1	41.4	ug/L	0.065	0.2	
6090602-ICV1	Ti Total 6020	43.27606		ug/L	1	43.3	ug/L	0.034	0.2	
6090602-ICV1	V Total 6020	40.95888		ug/L	1	41.0	ug/L	0.17	0.2	
6090602-SCV1	Ag Total 6020	42.35505		ug/L	1	42.4	ug/L	0.024	0.04	
6090602-SCV1	Cu Total 6020	42.45288		ug/L	1	42.5	ug/L	0.065	0.2	
6090602-SCV1	Pb Total 6020	41.42369		ug/L	1	41.4	ug/L	0.065	0.2	
6090602-SCV1	Ti Total 6020	43.27606		ug/L	1	43.3	ug/L	0.034	0.2	
6090602-SCV1	V Total 6020	40.95888		ug/L	1	41.0	ug/L	0.17	0.2	
6090602-ICB1	Ag Total 6020	0.009002		ug/L	1	0.00900	ug/L	0.024	0.04	
6090602-ICB1	Cu Total 6020	0.004933		ug/L	1	0.00493	ug/L	0.065	0.2	
6090602-ICB1	Pb Total 6020	0.003567		ug/L	1	0.00356	ug/L	0.065	0.2	
6090602-ICB1	Ti Total 6020	0.004911		ug/L	1	0.00491	ug/L	0.034	0.2	
6090602-ICB1	V Total 6020	0.016471		ug/L	1	0.0165	ug/L	0.17	0.2	
6090602-CRL1	Ag Total 6020	0.224835		ug/L	1	0.225	ug/L	0.024	0.04	
6090602-CRL2	Cu Total 6020	1.146779		ug/L	1	1.15	ug/L	0.065	0.2	

TriMatrix Laboratories, Inc.

Data Review Report -- Metals Lab  
Sequence = 6090602 Page 4 of 4

SampleID	Analyte	IRResult	Qualifier	Units	Diln	EResult	EUnits	IMDL	IMDL	EM
6090367-02	Cu Total 6020	2.761238	ME004	ug/L	1	14	ug/L	0.322	4	4
6090367-02	Ag Total 6020	0.020662		ug/L	1	0.10	ug/L	0.0726	0.2	0.2
6090367-03	V Total 6020	0.947256		ug/L	1	4.7	ug/L	0.259	4	4
6090367-03	Cu Total 6020	0.817267		ug/L	1	4.1	ug/L	0.322	4	4
6090367-03	Ag Total 6020	0.010797		ug/L	1	0.054	ug/L	0.0726	0.2	0.2
6090602-CCV4	Ag Total 6020	43.3769		ug/L	1	43.4	ug/L	0.024	0.04	
6090602-CCV4	Cu Total 6020	43.01479		ug/L	1	43.0	ug/L	0.065	0.2	
6090602-CCV4	V Total 6020	42.85077		ug/L	1	42.9	ug/L	0.17	0.2	
6090602-CCB4	Ag Total 6020	0.007124		ug/L	1	0.00712	ug/L	0.024	0.04	
6090602-CCB4	Cu Total 6020	0.007899		ug/L	1	0.00790	ug/L	0.065	0.2	
6090602-CCB4	V Total 6020	0.009901		ug/L	1	0.00990	ug/L	0.17	0.2	

Approved By:  2-22-10  
QA Officer

Approved By:  2-22-10  
Area Supervisor





SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: GR-01-129

Revision Number: 3.9  
Date Revised: 2/22/10  
Date Initiated: 12/4/97

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Attachment 23.5  
Internal Chain Of Custody Example

24-JUL-2000

TRIMATRIX LABORATORIES, INC.  
CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

PAGE 119

CLIENT: [REDACTED]

PROJECT: [REDACTED]

SUBMITTAL: Sampling Event #1

PROJECT: 35465-1

Parameter: ~~ARSENIC - TOTAL~~ All Metals

Method: MS/EPA/UT

Ref Clt: EPA-200.8/6020

Matrix: WATER

SAMPLE #	REMOVED BY: (SIGNATURE)	DATE & TIME REMOVED	RELINQUISHED BY: DATE & TIME	RECEIVED BY: DATE & TIME	DATE & TIME RETURNED
253868	<i>[Signature]</i>			7-21-00 <sup>SA</sup>	7-28-00 <sup>SP</sup>
253869					
253870					
253871					
253872					DSC
253873				DSC	
253874					
253875					
253876	<i>[Signature]</i>				
253877					
253878					
253879					
253880					
253881					
253882					
253883	<i>[Signature]</i>			7-21-00 <sup>SA</sup>	7-28-00 <sup>SP</sup>

Approved By: *[Signature]* 2-22-00  
QA Officer

Approved By: *[Signature]* 2-22-00  
Area Supervisor





SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
Perkin Elmer Elan 6000/6100  
SW-846 Method 6020A  
SOP Number: **GR-01-129**

Revision Number: 3.9  
Date Revised: 2/22/10  
Date Initiated: 12/4/97

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**Attachment 23.6**  
**SCV Standard Solution**

Element	Concentration (mg/L)	Diluted Concentration (ug/L)
Aluminum	10	40
Antimony	10	40
Arsenic	10	40
Barium	10	40
Beryllium	10	40
Boron	10	40
Cadmium	10	40
Chromium	10	40
Cobalt	10	40
Copper	10	40
Lead	10	40
Manganese	10	40
Molybdenum	10	40
Nickel	10	40
Selenium	10	40
Silver	10	40
Thallium	10	40
Tin	10	40
Vanadium	10	40
Zinc	10	40
Uranium	10	40
Strontium	10	40

Approved By: ml 2-22-10  
QA Officer

Approved By: He 2-22-10  
Area Supervisor



SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
 Perkin Elmer Elan 6000/6100  
 SW-846 Method 6020A  
 SOP Number: **GR-01-129**

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Revision Number: 3.9  
 Date Revised: 2/22/10  
 Date Initiated: 12/4/97

**Attachment 23.7  
 Element Conditions**

Int Std	Analyte	Mass [amu]	Scan Mode	MCA Channels	Dwell Time per AMU [ms]	Integration Time [ms]	Corrections
Li		6.0151	Peak Hopping	1	100	1000	
	Be	9.0122	Peak Hopping	1	100	1000	
		44.9559	Peak Hopping	1	100	1000	
	V	50.944	Peak Hopping	1	100	1000	C10, Cr
	Cr	52.9407	Peak Hopping	1	100	1000	
	Mn	51.9405	Peak Hopping	1	100	1000	
	Co	58.9332	Peak Hopping	1	100	1000	
	+Ni	59.9332	Peak Hopping	1	100	1000	
	Ni	60.931	Peak Hopping	1	100	1000	
	+Cu	62.9298	Peak Hopping	1	100	1000	
	Cu	64.9278	Peak Hopping	1	100	1000	
	+Zn	65.926	Peak Hopping	1	100	1000	
	Zn	66.9271	Peak Hopping	1	100	1000	
	Zn	67.9249	Peak Hopping	1	100	1000	
	As	74.9216	Peak Hopping	1	100	1000	ArCl, Se
	Se	81.9767	Peak Hopping	1	100	1000	
	Ge	88.9054	Peak Hopping	1	100	1000	
Mo	Mo	97.9055	Peak Hopping	1	100	1000	Ru
	+Ag	106.905	Peak Hopping	1	100	1000	
	Ag	108.905	Peak Hopping	1	100	1000	
	+Cd	110.904	Peak Hopping	1	100	1000	Mo, Pd
	Cd	113.904	Peak Hopping	1	100	1000	Sn
In	In	114.904	Peak Hopping	1	100	1000	Sn
	Sb	120.904	Peak Hopping	1	100	1000	
	+Sb	122.904	Peak Hopping	1	100	1000	Te
	Ba	134.906	Peak Hopping	1	100	1000	
	+Ba	136.905	Peak Hopping	1	100	1000	
Tb	Tb	158.925	Peak Hopping	1	100	1000	
	Tl	202.972	Peak Hopping	1	100	1000	
	+Tl	204.975	Peak Hopping	1	100	1000	
	Pb	207.977	Peak Hopping	1	100	1000	Pb, Pb
Bi		208.980	Peak Hopping	1	100	1000	

Approved By: PD 2-22-10  
 QA Officer

Approved By: OK 2-22-10  
 Area Supervisor



SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
 Perkin Elmer Elan 6000/6100  
 SW-846 Method 6020A  
 SOP Number: **GR-01-129**

Revision Number: 3.9  
 Date Revised: 2/22/10  
 Date Initiated: 12/4/97

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**Attachment 23.8**  
**Spiking Solutions Final Concentrations**

Element	Aqueous Samples (ug/L)	Solid Samples (mg/kg)
Aluminum	50	20
Antimony	50	20
Arsenic	50	20
Barium	50	20
Beryllium	50	20
Boron	50	20
Cadmium	50	20
Chromium	50	20
Cobalt	50	20
Copper	50	20
Lead	50	20
Manganese	50	20
Molybdenum	50	20
Nickel	50	20
Selenium	50	20
Silver	50	20
Thallium	50	20
Tin	50	20
Vanadium	50	20
Zinc	50	20

Approved By: ms 2-22-10  
 QA Officer

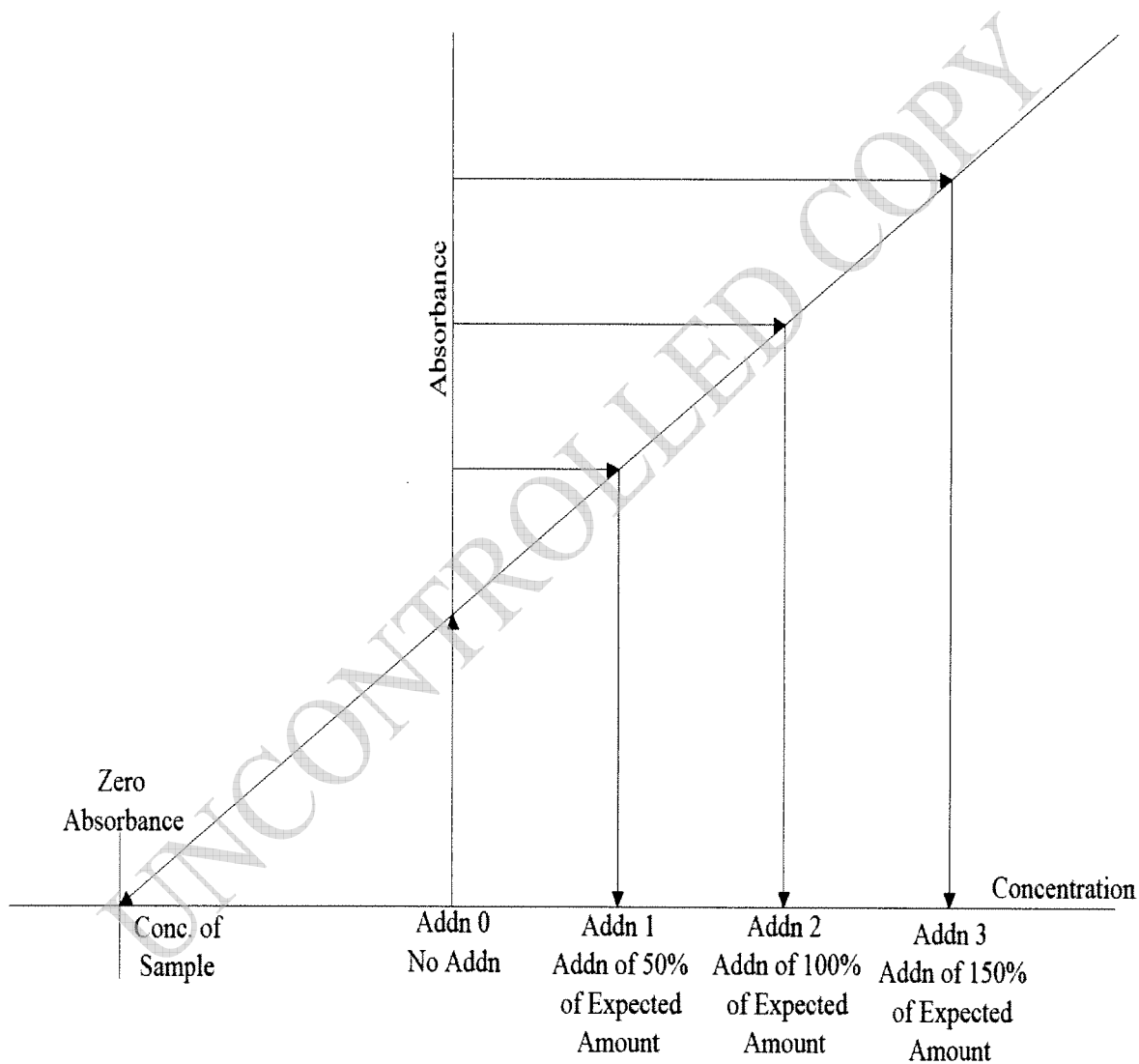
Approved By: OSC 2-22-10  
 Area Supervisor



SOP Name: Inductively Coupled Plasma - Mass Spectrometry Perkin Elmer Elan 6000/6100 SW-846 Method 6020A SOP Number: <b>GR-01-129</b>	Revision Number: 3.9  Date Revised: 2/22/10 Date Initiated: 12/4/97
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**Attachment 23.9**  
**MSA Curve Example**



Approved By: <u>          <i>aj</i> 2-22-10          </u> QA Officer	Approved By: <u>          <i>de</i> 2-22-10          </u> Area Supervisor
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SOP Name: Inductively Coupled Plasma - Mass Spectrometry  
 Perkin Elmer Elan 6000/6100  
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**Attachment 23.10**  
**Ionic Interference Correction Report Example**

## Quantitative Analysis Calibration Report

File Name:

File Path:

Calibration Type: External Calibration

Analyte	Mass	Curve Type	Slope	Intercept	Corr. Coeff.
---------	------	------------	-------	-----------	--------------

### Method Equation

Analyte	Mass	Corrections
V	50.944	-2.706684618 *(ClO 53 -(0.393948145 *Cr 52))
As	74.922	-2.706684618 *(ArCl 77 -(1.549850256 *Se 82))
Se	81.917	- 1.007833 *Kr 83
Mo	97.906	- 0.109613 *Ru 101
Cd	110.904	-0.909241101 *(MoO 108 -(0.712*Pd 106))
Cd	113.904	- 0.027250 * Sn 118
In	114.904	- 0.014038 * Sn 118
Sn	119.902	- 0.012730 * Te 125
Sb	122.904	- 0.125884 * Te 125
Pb	207.977	+1*Pb 206 +1*Pb 207

Approved By: MS 2-22-10  
 QA Officer

Approved By: Dec 2-22-10  
 Area Supervisor





## STANDARD OPERATING PROCEDURE

### Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1  
SW-846 Method 7470A

#### APPROVALS:

Area Supervisor: Marge A. Scott Date: 01-23-09  
Marge A. Scott

QA Officer: Tom C. Booher Date: 1-22-9  
Tom C. Booher

Operations Manager: Jeff P. Glaser Date: 1/23/09  
Jeff P. Glaser

Procedure Number: GR-01-140  
Revision Number: 0.4

Date Initiated: 2/19/03  
Effective Date: 2/20/09

Date Revised: 1/22/09  
Pages Revised: All

By: Marge A. Scott  
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>04-09-10</u>	<u>Marge A. Scott</u>	<u>04-09-11</u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>



SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
SW-846 Method 7470A, EPA Method 245.1  
SOP Number: **GR-01-140**

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Revision Number: 0.4  
Date Revised: 1/22/09  
Date Initiated: 2/19/03

## 1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.
- 1.2 The minimum reporting limit is 0.2 ug/L.

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"*
- 2.2 *Methods for the Determination of Metals in Environmental Samples, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994*

## 3.0 SUMMARY OF PROCEDURE


- 3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.
- 3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.
- 3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.
- 3.4 Inorganic mercury is converted to mercury in the Hg<sup>2+</sup> state during the digestion, for detection and quantitation.


## 4.0 PARAMETER OR COMPOUND LIST

- 4.1 Mercury

## 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision
- 5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

Approved By:  1-22-09  
QA Officer

Approved By:  01-23-09  
Area Supervisor



SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
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5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Action*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

## 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil often contains mercury at low levels. When soil samples are mixed and ground, liquid samples may become contaminated. Therefore, keep the preparation area for aqueous mercury samples free of dust and dirt to avoid cross-contamination.

6.2 Also, areas where Kjeldahl nitrogen (TKN) and/or chemical oxygen demand (COD) samples are being prepared and analyzed can be sources of contamination if not properly coordinated.

6.3 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory.

6.4 Mercury may be lost if the block digester temperature exceeds 95° C. Use a digital thermometer to monitor block digestion temperature, to prevent mercury loss from boiling. Record block digester temperatures on the pre-treatment benchsheet.

6.5 Reagents may become contaminated over time. If contamination is suspected before a reagent's assigned expiration date, discard it and acquire or prepare fresh reagent.

6.6 Volatile materials such as chlorine which absorb at 253.7 nm are a positive interference. If volatile gases are suspect, notify the instrumental analyst to purge the dead air space in each digestion cup with an inert gas before adding stannous chloride solution.

6.7 Sulfide, chloride, copper (>10 mg/L) and tellurium are interferences.

6.8 Organic compounds which absorb around 253.7 nm are interferences. Such interferant concentration levels are difficult to isolate and quality control must be strictly followed to minimize.

## 7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids and oxidizers are used in this procedure. Wear disposable gloves, approved safety glasses and appropriate protective clothing at all times when handling these and other reagents. Always wear disposable gloves when handling digestates as well. Refer to the MSDS library on the laboratory intranet library for information on these or other chemicals utilized in the laboratory.

7.3 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate any instrument if it is not properly vented. Mercury can also be absorbed through the skin. Wear

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QA Officer

Approved By:     MAS    01-23-09      
Area Supervisor



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disposable gloves at all times when handling laboratory chemicals. Elemental Hg and  $\text{Hg}^{2+}$  are toxic if ingested. Always wash hands after handling mercury or any other chemical used in the laboratory.

- 7.4 No food or drink is allowed in the metals laboratory or any other laboratory within the facility. Food and drink may be consumed in laboratory offices if separated from the laboratory but may only be stored in the facility cafeteria.
- 7.5 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the metals laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed to a third party through casual contact.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

- 8.1 Samples must be acidified at the time of collection to a pH of  $<2$ . If the pH is not  $<2$  when received by the laboratory, lower to  $<2$  during the sample receipt process using 1:1 nitric acid/water. The maximum volumes of acid added to a sample (including initial volume) are:


Container Size	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10.0

NOTE: Sample preservation must occur in the original container to prevent the loss of any analytes adsorbed to the container wall. It is not permissible to remove and acidify a sample aliquot.

- 8.2 If a pH adjustment is necessary, a minimum of 24 hours must elapse before the sample digestion process may begin.
- 8.3 Consult the project chemist for instruction if the pH cannot be adjusted to  $<2$  using the specified maximum amount of acid. Samples prepared and analyzed without proper pH preservation, results must be narrated as such.
- 8.4 Samples may be collected in glass or plastic. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.
- 8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 550 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.
- 8.6 Properly acid-preserved samples can be held for up to 28 days prior to digestion. Refrigeration is not required for samples or digestates.

## 9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Micropipette, capable of delivering 100 to 1000  $\mu\text{L}$

Approved By:  1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor



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- 9.2 HotBlock™ block digester, Environmental Express, model SC154
- 9.3 Polypropylene Digestion vessels certified pre-cleaned and pre-calibrated to 50 mL, Environmental Express, catalog SC-475
- 9.4 Block digester cover
- 9.5 Filtermate, Environmental Express, catalog SC0501
- 9.6 Carboy, 50 L, high density polyethylene, with spigot
- 9.7 Beaker, 1 L, polyethylene
- 9.8 Volumetric flasks, type A, glass, 100 mL

## 10.0 ROUTINE PREVENTIVE MAINTENANCE

- 10.1 Verify and document micropipette calibration daily. Refer to TriMatrix GR-10-111 for micropipette calibration instructions.
- 10.2 Clean the block digester and counters daily, and when spills and/or splattering occur.

## 11.0 CHEMICALS AND REAGENTS

- 11.1 Concentrated nitric acid (HNO<sub>3</sub>), trace-metal grade
- 11.2 Concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), trace-metal grade
- 11.3 Metals laboratory reagent water
  - 11.3.1 ASTM Type II Milli-Q system water is fed into an all-glass Barnstead distillation unit. The distillate is immediately stored in a 50 L high-density polyethylene carboy.
  - 11.3.2 This is the only water acceptable for use in the metals laboratory and will be designated as metals laboratory reagent water.
- 11.4 Potassium persulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>), ACS grade or equivalent, prepare a 5% (w/v) solution as follows:
  - 11.4.1 Weigh out 25 g ACS-grade K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> into a 1 L polyethylene bottle. Add 500 ml distilled reagent water to the beaker. Place a stir bar in the solution, cover and stir on a magnetic stir plate until all crystals have dissolved.
  - 11.4.2 This solution takes several hours to prepare. Keep the reagent in a tightly capped bottle when not in use to avoid contamination. Prepare fresh weekly or sooner if a precipitate forms.

Approved By: OR 1-22-09  
 QA Officer

Approved By: UAS 01-23-09  
 Area Supervisor





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11.5 Potassium permanganate ( $\text{KMnO}_4$ ), low-mercury ACS grade, prepare a 5% solution (w/v) as follows:

11.5.1 Weigh out and transfer 25 g  $\text{KMnO}_4$  crystals to a 1 L polyethylene bottle. Add 500 ml metals laboratory reagent water to the container. Place a stir bar in the beaker then cover and stir on a magnetic stir plate until all crystals are dissolved. Since this solution is nearly saturated it may take several hours to prepare. Prepare fresh daily.

11.5.2 Update the standard number in Element™ when a new lot number of  $\text{KMnO}_4$  is used in the preparation.

11.5.3 If  $\text{KMnO}_4$  solution is spilled, immediately soak up with absorbent pads or paper towels then clean with water. Place all contaminated pads or towels in a plastic bag to be appropriately disposed of. If a brownish stain remains from the spill, carefully remove by applying hydroxylamine hydrochloride. Prevent exposure to the skin and eyes at all times by wearing disposable gloves, safety glasses and a laboratory coat.

11.5.4 Potassium permanganate absorbs mercury when exposed to laboratory air. Keep the reagent bottle tightly sealed when not in use. Store in a dark cabinet to minimize autocatalytic reduction.

11.6 Sodium chloride ( $\text{NaCl}$ )/hydroxylamine hydrochloride ( $\text{NH}_2\text{OH}\cdot\text{HCl}$ ), prepare a 12% solution (w/v) as follows:

11.6.1 Weigh out and transfer 30 g of  $\text{NaCl}$  and 30 g of  $\text{NH}_2\text{OH}\cdot\text{HCl}$  to a 500 mL polyethylene bottle. Add 250 mL of distilled reagent water and a stir bar.

11.6.2 Place the container on a stir plate and stir until all crystals are dissolved. Prepare fresh daily.

11.6.3 Update the standard number in Element™ when a new lot number of either  $\text{NaCl}$  or  $\text{NH}_2\text{OH}\cdot\text{HCl}$  is used in the preparation.

## 12.0 STANDARDS PREPARATION

12.1 Mercury stock standard is purchased commercially at 1000 mg/L from Inorganic Ventures. The second-source secondary calibration verification (SCV) standard is purchased at 1,000 mg/L from SCP Science.

12.1.1 Purchased mercury solutions have an expiration date of the date specified by the manufacturer.

12.1.2 Log each standard into Element™.

12.1.3 All stock standards are single-element solutions.

12.2 Intermediate standard (1000 ug/L mercury)

Approved By: JS 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor





Laboratories, Inc.

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### 12.2 Intermediate standard (1000 ug/L mercury)

12.2.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.

12.2.2 Add about 20 mL of metals laboratory reagent water to the flask.

12.2.3 Add 0.5 mL concentrated trace-metal grade  $\text{HNO}_3$  then swirl to mix.

12.2.4 Pipette 100  $\mu$ L of 1000 mg/L mercury standard into the flask.

12.2.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.

12.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.3 Prepare calibration standards as follows:

12.3.1 Prepare 0 ug/L (a blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 2.0 ug/L, and 5.0 ug/L mercury standards. Begin the preparation by labeling six 100 mL volumetric flasks. Rinse the flasks several times with metals laboratory reagent water.

12.3.2 Add approximately 20 mL metals laboratory reagent water to each flask.

12.3.3 Add 0.5 mL concentrated trace-metal grade  $\text{HNO}_3$  to each flask and swirl.

12.3.4 Pipette 500  $\mu\text{L}$  of 1000 $\mu\text{g/L}$  Hg standard from Section 12.3 into the vessel labeled “5.0  $\mu\text{g/L}$  Hg”. Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.5 Pipette 200  $\mu\text{L}$  of 1000  $\mu\text{g/L}$  Hg standard from Section 12.3 into the vessel labeled “2.0  $\mu\text{g/L}$  Hg”. Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.6 Pipette 100  $\mu\text{L}$  of 1000  $\mu\text{g/L}$  Hg standard from Section 12.3 into the vessel labeled “1.0  $\mu\text{g/L}$  Hg”. Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.7 Pipette 50 µL of the 1000 ug/L Hg standard from Section 12.3 into the vessel labeled “0.5 ug/L Hg”. Dilute to volume with metals laboratory water then cap and mix.

12.3.8 Pipette 20 µL of the 1000 µg/L Hg standard from Section 12.3 into the vessel labeled “0.2 µg/L Hg”. Dilute to volume with metals laboratory water then cap and mix.

12.3.9 Dilute the volumetric labeled “Hg blank” to volume with metals laboratory water then cap and mix.

12.9.10 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.4 Prepare the Secondary Source Verification (SCV) as follows:

Approved By:                      1-22-09  
QA Officer

Approved By: WAS 01-23-09  
Area Supervisor



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12.4.1 Intermediate SCV standard (1000 ug/L mercury)

- 12.4.1.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.
- 12.4.1.2 Add about 20 mL of metals laboratory reagent water to the flask.
- 12.4.1.3 Add 0.5 mL concentrated trace-metal grade  $\text{HNO}_3$  then swirl to mix.
- 12.4.1.4 Pipette 100  $\mu\text{L}$  of 1000 mg/L second-source mercury standard into the flask.
- 12.4.1.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.
- 12.4.1.6 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.4.2 Prepare the secondary calibration verification (SCV) standard as follows:

- 12.4.2.1 Label a 100 mL volumetric flask as “3.0 ug/L SCV Hg”. Rinse the flask several times with metals laboratory reagent water.
- 12.4.2.2 Add approximately 20 mL metals laboratory reagent water.
- 12.4.2.3 Add 0.5 mL concentrated trace-metal grade  $\text{HNO}_3$  to each flask and swirl.
- 12.4.2.4 Pipette 0.30 mL of 1000 ug/L Hg standard from Section 12.4.1 into the volumetric. Dilute to volume with metals laboratory reagent water then cap and mix.
- 12.4.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.5 Complete preparation of the calibration and SCV standards as follows:

- 12.5.1 Measure 30 mL of each calibration standard and the SCV into digestion vessels.
- 12.5.2 Pipette 0.75 mL of concentrated  $\text{HNO}_3$  into each vessel.
- 12.5.3 Pipette 1.5 mL of concentrated  $\text{H}_2\text{SO}_4$  into each vessel.
- 12.5.4 Swirl to mix thoroughly.
- 12.5.5 Pipette 4.5 mL of 5%  $\text{KMnO}_4$  into each vessel and let stand for 15 minutes.
- 12.5.6 Pipette 2.4 mL of 5%  $\text{K}_2\text{S}_2\text{O}_8$  into each vessel.

Approved By: MA 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor



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- 12.5.8 Place in the block digester and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C.
- 12.5.7 After digesting, cool to room temperature then pipette 1.80 mL of 12% NaCl/NH<sub>2</sub>OH·HCl into each digestion vessel.
- 12.5.9 After adding, bring back to 50 mL with metals laboratory reagent water then cap tightly and mix thoroughly.
- 12.5.10 After a delay of at least 30 seconds, calibration standards and SCV are ready for the stannous chloride addition and immediate analysis.
- 12.6 Spike samples for quality control as follows:
- 12.6.1 Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 10 samples for each wastewater and/or potable water matrix run in accordance with method 245.1. Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 20 samples for each water matrix run in accordance with method 7470A..
- 12.6.2 Prepare 2.0 ug/L matrix spikes (MS/MSD) as follows:
- 12.6.2.1 Measure 30 mL of the sample being spiked into each of two block digestion vessels.
- 12.6.2.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into each vessel.
- 12.6.2.3 Prepare these spiked sample aliquots in the same way the un-spiked sample is prepared.
- 12.6.3 Prepare a 0.5 ug/L blank spike (BS) as follows:
- 12.6.3.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.
- 12.6.3.2 Pipette 15 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.
- 12.6.3.3 Prepare in the same way as a sample, bringing the final volume to 50 mL.
- 12.6.4 Prepare a 2.0 ug/L blank spike (BS) as follows:
- 12.6.4.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.
- 12.6.4.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.
- 12.6.4.3 Prepare in the same way as a sample bringing the final volume to 50 mL.

Approved By:           m          1-22-09            
QA Officer

Approved By:           MAS          01-23-09            
Area Supervisor



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### 13.0 ANALYTICAL PROCEDURE

- 13.1 All samples and quality control must be digested before analysis. All digestates must be evaporated to below 50 mL within the digestion time and brought back to a final volume of 50 mL with metal laboratory reagent water at the end of the digestion.
- 13.2 Pre-heat the block digester to 90-95° C.
- 13.3 Measure 30 mL of each well-mixed sample into a digestion vessel. Prepare the associated quality control samples and calibration standards.
- 13.4 To each digestion vessel add the following reagents:
- 13.4.1 Pipette in 0.75 mL of concentrated  $\text{HNO}_3$
  - 13.4.2 Pipette in 1.5 mL concentrated  $\text{H}_2\text{SO}_4$
  - 13.4.3 Pipette in 4.5 mL of  $\text{KMnO}_4$
- 13.5 After adding, swirl to mix then let the samples sit for 15 minutes.
- 13.5.1 If a sample does not maintain the purple or brown permanganate color for at least 15 minutes, add up to an additional 3.0 mL of  $\text{KMnO}_4$  solution but no more.  
  
Note: Separate calibration standards and quality control must be prepared with the same permanganate volume to match the sample.
  - 13.5.2 If a sample does not maintain the purple or brown permanganate color after the maximum  $\text{KMnO}_4$  addition of 7.5 mL, discard the preparation, dilute the sample appropriately and prepare 30 mL of the dilution for digestion.
- 13.6 Pipette 2.40 mL of 5%  $\text{K}_2\text{S}_2\text{O}_8$  into each digestion.
- 13.7 Place in the block digester and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C. Do not exceed 95° C.
- 13.8 After digestion, remove and cool to room temperature then pipette in 1.8 mL of 12%  $\text{NaCl}/\text{NH}_2\text{OH}\cdot\text{HCl}$ .
- 13.9 Finally, bring to a final volume of 50 mL with metals laboratory reagent water. Cap and mix thoroughly.
- 13.10 After a delay of at least 30 seconds, samples are ready for the stannous chloride addition and immediate analysis.

### 14.0 DATA REPORTING AND DELIVERABLES

Approved By: MS 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor



SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
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- 14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.
- 14.1.1 Initial and date any hand-written information on the work order report.
- 14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.
- 14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch benchsheet report is printed.
- 14.2.1 Enter all information associated with the digestion before printing.
- 14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.
- 14.2.3 Initial and date any hand-written information on the preparation batch report.
- 14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.
- 14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used. (Attachment 20.1)
- 14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data. (Attachment 20.3)
- 14.5 If internal chain-of-custody (COC) is required, it is very important that COC forms be filled in completely and correctly. Organize, transfer and/or archive COC forms in accordance with the TriMatrix quality control manual.
- 14.6 Fill in block digester maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.
- 14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

## 15.0 QUALITY ASSURANCE

- 15.1 Prepare and digest a method blank (BLK) for each batch of up to 20 samples digested, to monitor contamination. The BLK is 30 mL of metals laboratory reagent water measured into a block digestion vessel. The BLK vessel is then processed as a sample following every step in the procedure. Refer to TriMatrix SOP GR-01-123 for BLK acceptance criteria.
- 15.2 Prepare and digest the two blank spikes (BS) of Section 12.6.3 and 12.6.4 for each batch of up to 20 samples digested. Refer to TriMatrix SOP GR-01-123 for BS acceptance criteria.

Approved By: JA 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor



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- 15.3 Prepare and digest a matrix spike (MS)/matrix spike duplicate (MSD) as described in Section 12.6.2 and at the frequency described in Section 12.6.1, or prepare for each digestion session/matrix. Matrix spikes monitor matrix recovery and precision. Refer to TriMatrix SOP GR-01-123 for MS/MSD acceptance criteria.
- 15.4 Do not allow samples to boil during digestion. If a sample begins to boil, immediately remove it from the block digester and turn the temperature down.
- 15.5 Do not use expired spiking solutions and/or standards. If a standard has expired, obtain or prepare another.
- 15.6 The method of standard additions must be performed on all leachates with 50% or less spike recovery. Refer to TriMatrix SOP GR-01-123 for preparing and digesting a standard addition series.

## 16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

- 19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

### 19.1.1 Initial Demonstration of Capability

- 19.1.1.1 Prepare four blank spikes (BS) at a concentration of 2.0 ug/L in laboratory reagent water. ONLY the last four results may be used. Otherwise, digest and evaluate the four blank spikes following every step in the digestion and analysis procedures. Calculate average percent recovery and relative standard deviation by inputting the four result values to the IDC spreadsheet on the laboratory intranet library. Average percent recovery must fall within 85 – 115% recovery. Relative standard deviation must be  $\leq 20\%$ .

- 19.1.1.2 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the procedure or techniques used then repeat the study.


- 19.1.1.3 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed. Copies of successful study spreadsheets must be given to the quality assurance department.

### 19.1.2 Continuing Demonstration of Capability (CDC)

- 19.1.2.1 A demonstration of capability must be repeated annually by all analysts running samples.

- 19.1.2.2 The CDC may be accomplished by repeating the IDC study, by submitting four consecutive blanks spike values obtained during the course of routine sample

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QA Officer

Approved By:  01-23-09  
Area Supervisor



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digestion, by submitting the last four MDL study values or by submitting an acceptable performance testing result. In each case, the work must be done exclusively by one analyst and submitted to quality control for documentation.

- 19.2 A Method Detection Limit (MDL) Study must be completed annually in accordance with TriMatrix SOP GR-10-125.

## **17.0 POLLUTION PREVENTION**

- 17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.
- 17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.
- 17.3 Conserve the use of chemicals where applicable.
- 17.4 Comply with all environmental laws associated with chemicals in the laboratory.

## **18.0 WASTE MANAGEMENT**

- 18.1 Consult the appropriate Material Safety Data Sheet (MSDS) on the laboratory intranet library when disposing of chemicals.
- 18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.
- 18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

## **19.0 REFERENCES**

- 19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"*
- 19.2 *Methods for the Determination of Metals in Environmental Samples, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994*

## **20.0 ATTACHMENTS**

- 20.1 Mercury Stock Standard Certificate of Analysis Example
- 20.2 Element™ Work Order Report Example

Approved By: *m 1-22-09*  
QA Officer

Approved By: *MAS 01-23-09*  
Area Supervisor



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SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
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20.3 Element™ Preparation Batch Report Example

20.4 Stock Standards Log Example

20.5 Digestion Flowchart

UNCONTROLLED COPY

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**Attachment 20.1**  
**Mercury Stock Standard Certificate of Analysis Example**



**inorganic ventures / iv labs**

195 lehigh avenue, suite 4, lakewood, nj 08701 usa  
 phone: 800-669-6799 • 732-901-1900 • fax: 732-901-1903  
 e-mail: [ivsales@ivstandards.com](mailto:ivsales@ivstandards.com) • website: [www.ivstandards.com](http://www.ivstandards.com)

**certificate of analysis**

**1.0 Inorganic Ventures / IV Labs** is an ISO Guide 34-2000 Certified Reference Material (CRM) Manufacturer: **Certificate #883-02**. The certificate is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31-2000 (Reference Materials - Contents of certificates and label(s), ISO Guide 34-2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principles."

**2.0 DESCRIPTION OF CRM** Custom-Grade 1000 µg/mL Mercury in 3.5% (abs) HNO<sub>3</sub>

Catalog Number: CGHG1-1, CGHG1-2, and CGHG1-5  
 Lot Number: **Y-HG02038**  
 Starting Material: Hg metal  
 Starting Material Purity (%): 99.999555  
 Starting Material Lot No: 05214TX  
 Matrix: 3.5% (abs) HNO<sub>3</sub>

*me 14.98.8*  
*AS03277*  
*OK*  
*9-27-05*

**3.0 CERTIFIED VALUES AND UNCERTAINTIES**

**Certified Concentration:** 1007 ± 3 µg/mL

**Certified Density:** 1.026 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$$

$$\text{Uncertainty } (\pm) = 2 \left( \frac{\sum \sigma_i^2}{n} \right)^{1/2}$$

( $\bar{x}$ ) = mean  
 $x_i$  = individual results  
 $n$  = number of measurements  
 $\sum \sigma_i^2$  = The summation of all significant estimated errors.  
 (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

**4.0 TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 8.10)

This IV product is Traceable to NIST via direct comparison to NIST SRMs. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors.

**4.1 Assay Method #1** **998 ± 4 µg/mL**  
 EDTA NIST SRM 928 Lot Number: 392110  
**Assay Method #2** **1007 ± 3 µg/mL**  
 ICP Assay NIST SRM 3133 Lot Number: 991304

Approved By: MD 1-22-09  
 QA Officer

Approved By: MAS 01-23-09  
 Area Supervisor





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Attachment 20.2  
Element™ Work Order Report Example

TriMatrix Laboratories, Inc.

**WORK ORDER 0610156**

Printed: 10/12/2006 2:46:21PM

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## Metals Sample Receipt Notice

Client: [REDACTED]	Project Manager: Gary L. Wood
Project: [REDACTED]	Project Number: [none]
Client Due Date: Oct-20-06 16:00 (10 day TAT)	Report Level: 2RL
W.O. Comments: 2RL	

Lab Number	Sample Name Analysis	Matrix	Sampled Date		Sample Comments	
			TAT	Expire Date	Lab Due Date	Comments
	Ag Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	As Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Ba Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Cd Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Cr Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Cu Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Hg Total 7470A	10	Nov-03-06 14:00	Oct-19-06 17:00		
	Ni Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Pb Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Se Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		
	Zn Total 6020	10	Apr-04-07 14:00	Oct-19-06 17:00		

Approved By: gr 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor





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**Attachment 20.3**  
**Element™ Preparation Batch Report Example**

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0901183** Page 1 of 1

Printed: 1/22/2009 1:38:35PM

Metals, Water, 7470A Digestion - Total

(No Surrogate)

Batch Comments: MAS 01/21/09 1:00-3:00PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0901183	Hg Total 7470A				

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client QC Type	Extraction Comments
0901183-BLK1		Jan-21-09 13:00	MAS	30	30					BLANK	
0901183-BS1		Jan-21-09 13:00	MAS	30	30			8100309	60	LCS	
0901183-MS1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE	
0901183-MSD1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE DUP	
0901199-10	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-11	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-12	E	Jan-21-09 13:00	MAS	30	30						wtr
0901199-13	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-14	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-15	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-16	C	Jan-21-09 13:00	MAS	30	30						wtr
0901199-17	C	Jan-21-09 13:00	MAS	30	30						wtr

Approved By: MD 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor



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**Attachment 20.4**  
**Stock Standards Log Example**

**Analytical Standard Record**

**TriMatrix Laboratories, Inc.**

**8060501**

Description:	Mercury 1000 mg/L SCV Stock	Expires:	Feb-28-10
Standard Type:	Other	Prepared:	Jun-17-08
Solvent:	10% HNO <sub>3</sub>	Prepared By:	** Vendor **
Final Volume (mls):	100	Department:	Metals
Vials:	1	Last Edit:	Jun-17-08 12:56 by RGJ

SCP part= 140-051-801  
 Lot=SC8122815  
 Received 06/17/08

Analyte	CAS Number	Concentration	Units
Mercury, OWEP-TAR	7439-97-6	1000	mg/L
Mercury, OWEP-SAR	7439-97-6	1000	mg/L
Mercury	7439-97-6	1000	mg/L

Approved By: MB 1-22-09  
 QA Officer

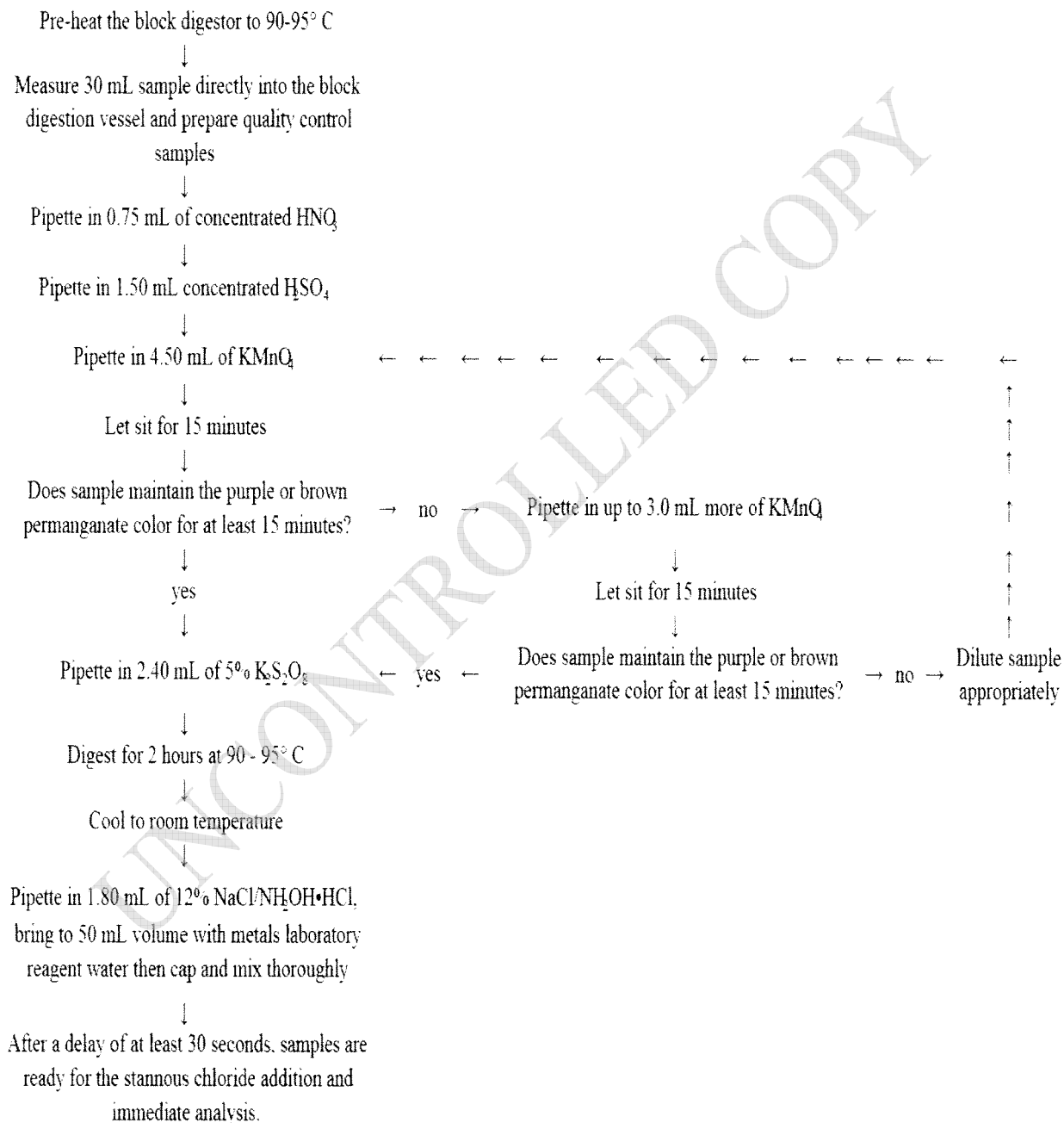
Approved By: MAS 01-23-09  
 Area Supervisor



SOP Name: Digestion of Mercury in Water, Wastewater and Aqueous Waste  
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**Attachment 20.5**  
**Digestion Flowchart**



Approved By: MA 1-22-09  
QA Officer

Approved By: MAS 01-23-09  
Area Supervisor





## STANDARD OPERATING PROCEDURE

### Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS

#### SW-846 Method 3020A

#### APPROVALS:

Area Supervisor: Marge A. Scott Date: 01-14-09  
Marge A. Scott

QA Officer: Tom C. Boocher Date: 1-8-09  
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/15/09  
Jeff P. Glaser

Procedure Number: GR-01-148  
Revision Number: 0.3

Date Initiated: 12/3/01  
Effective Date: 1/20/09

Date Revised: 1/7/09  
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 13

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>04-09-10</u>	<u>Marge A. Scott</u>	<u>04-09-11</u>
_____	_____	_____
_____	_____	_____



SOP Name: Block Digestion of Aqueous Samples and Extracts for  
Total/Dissolved Metals by ICPMS  
SW-846 Method 3020A  
SOP Number: **GR-01-148**

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Revision Number: 0.3  
Date Revised: 1/7/09  
Date Initiated: 12/3/01

## 1.0 SCOPE AND APPLICATION

- 1.1 This procedure outlines total metals digestion in water and leachate samples with the exception of silica-based matrices. Also outlined is the preparation of aqueous samples for dissolved metals analysis. A block digestion system is used when digestion is required.
- 1.2 Samples that have been filtered and acidified for dissolved metals analysis do not need the acid digestion unless otherwise specified.
- 1.2 The digestion is for analysis by inductively coupled plasma/mass spectrum (ICP/MS).

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update III, Revision 1, July 1992, Method 3020A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy"*

## 3.0 SUMMARY OF PROCEDURE

- 3.1 A representative sample aliquot is refluxed in nitric acid until the solution is light in color or the color remains stable. The digestate is then diluted to a predetermined volume for analysis.
- 3.2 Deviation from the referenced method involves sample volume reduction from 100 to 25 mL, with nitric acid additions adjusted in direct proportion to the volume reduced.

## 4.0 PARAMETER OR COMPOUND LIST

- 4.1 Refer to Attachment 20.1.

## 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.2 TriMatrix SOP GR-16-103, *Glassware Cleaning and Preparation for the Wet Chemistry and Metals Laboratories*, latest revision
- 5.3 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision
- 5.4 TriMatrix SOP GR-10-111, *Fixed and Adjustable Mechanical Pipette and Bottle Top Dispenser Calibration Verification*, latest revision
- 5.5 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

Approved By: POS 1-8-09  
QA Officer

Approved By: NAS 01-14-09  
Area Supervisor



SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A	Revision Number: 0.3
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5.6 TriMatrix SOP GR-10-106, Inorganic and Metals Laboratories Corrective Actions, latest revision

## 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples must be at room temperature before a digestion aliquot can be taken to assure measurement accuracy.

## 7.0 SAFETY PRECAUTIONS

7.1 Refer to the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan for routine precautions.

7.2 Gloves, approved safety glasses and laboratory coats must be worn at all times when handling acids and when performing this procedure. An impervious plastic apron is also required when handling significant volumes of acid.

7.3 Concentrated nitric acid fumes burn, discolor the skin and give off poisonous gases. Fume hoods must be used during handling and when performing this procedure.

7.4 Rinse all micropipette tips and beakers before cleaning or disposal. Do not dispose of acid rinses before neutralizing.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND MATERIALS

8.1 Samples must be acidified at the time of collection to a pH of <2 with nitric acid. If the pH is not <2 when received by the laboratory, lower to <2 during sample receipt using 1:1 nitric acid/water. The maximum allowable acid volumes that may be added to a sample (including initial volume) are:

Container Size (mL)	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10

Note: Sample preservation must occur in the original container to prevent analyte loss from plating out on the container wall. It is not permissible to remove and acidify a sample aliquot.

8.2 If pH adjustment is necessary when received at the laboratory, a minimum of 24 hours must elapse before the sample may be digested.

Approved By: W 1-5-09 QA Officer      Approved By: KAS 01-14-09 Area Supervisor



SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A SOP Number: <b>GR-01-148</b>	Revision Number: 0.3  Date Revised: 1/7/09 Date Initiated: 12/3/01
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- 8.3 Consult the project chemist for instructions if the pH cannot be adjusted to <2 using the maximum acid volume. Samples digested and analyzed without proper pH preservation, results must be narrated as such.
- 8.4 Samples must be collected in plastic. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.
- 8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 500 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.
- 8.6 Samples collected for dissolved metals analysis must be filtered and the filtrate acidified at the time of collection. For some sample determinations of dissolved elements, the filtered/acidified filtrate may require digestion.
- 8.7 Appropriately acid-preserved samples may be held for up to 180 days prior to digestion. Refrigeration is not required for samples or digestates.

## 9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

- 9.1 Micropipetter – capable of delivering 25 and 100 uL
- 9.2 Block digester for metals digestion – Environmental Express HotBlock™
- 9.3 Digestion cups, polypropylene, certified precleaned – Environmental Express SC499 or SC500
- 9.4 Ribbed watch glasses – Environmental Express SC505
- 9.5 Filtermate (digestate filtration) – Environmental Express SC0501

Note: If digestates are filtered after digestion, all associated quality control must be filtered as well.

- 9.6 Nonribbed watch glasses – SCP Science 010-500-081
- 9.7 Filtration units, vacuum pump, for dissolved sample filtering in the field
- Note: Each lot of filtration units received must be verified by running a dissolved blank and a dissolved blank spike. Results must be uploaded to Element™.

## 10.0 ROUTINE PREVENTIVE MAINTENANCE

- 10.1 Verify and document micropipette calibration daily.
- 10.2 Clean block digestion units and counters daily and/or when spills occur.

## 11.0 CHEMICALS AND REAGENTS

Approved By: <u>MA 1-8-09</u> QA Officer	Approved By: <u>MAS 01-14-09</u> Area Supervisor
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SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A	Revision Number: 0.3
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11.1 Laboratory distilled, deionized water (ASTM Type II) - designated in this procedure as metals-free water

11.2 Concentrated nitric acid (HNO<sub>3</sub>), trace metal grade

## 12.0 STANDARDS PREPARATION

12.1 Multi-element ICPMS certified spiking standards are listed in Attachment 20.1.

## 13.0 ANALYTICAL PROCEDURE

13.1 Prepare samples that have been filtered through a 0.45 µm filter and acidified in the field as follows:

13.1.1 If a filtered/acidified sample exhibits a strong organic odor, digest before analysis.

Note: Do not directly smell any sample.

13.1.2 If a filtered/acidified sample exhibits significant color, digest before analysis.

13.1.3 If a filtered/acidified sample has a turbidity greater than 1 NTU, digest before analysis.

13.1.4 If a filtered/acidified sample forms a precipitate at preservation, during transit to the laboratory or in storage, digest before analysis.

13.2 To perform the digestion, mix samples by shaking vigorously to achieve homogeneity. After mixing, measure 25.0 mL of sample (or an appropriate dilution) into a polypropylene digestion cup using the cup graduations. For samples requiring matrix spikes, shake again before subsequent 25.0 mL transfers.

Note: Each digestion cup lot needs verified for acceptable accuracy at 25 mL by weighing out 25 mL of metals-free water on the analytical balance. Then, calculate the average volume measured and uncertainty.

13.3 Add 0.75 mL concentrated HNO<sub>3</sub>, cover each cup with a ribbed watchglass and reflux in the block digester at 90 - 95° C. Adjust the block digester temperature to 85° C before refluxing to account for the watchglass, which adds ten degrees. Refer to the HotBlock™ operating manual for temperature adjustment and principles of operation. Evaporate to near dryness which is approximately 1.25 mL but don't let any digestion go completely dry.

13.4 After reducing to about 1.25 mL, remove from the block digester and cool to room temperature.

13.5 Add 0.75 mL concentrated HNO<sub>3</sub>, cover with a nonribbed watchglass and gently reflux for 30 minutes. Repeat with additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

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Approved By:           m j f 09            
QA Officer

Approved By:           WAS 01-14-09            
Area Supervisor



SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A SOP Number: <b>GR-01-148</b>	Revision Number: 0.3  Date Revised: 1/7/09 Date Initiated: 12/3/01
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- 13.6 When digestion is complete, evaporate to 0.75 mL using a ribbed watchglass. Do not let any part of the digestion cup bottom evaporate to dryness. If a digestate evaporates to dryness, it must be repeated.
- 13.7 Remove from the block digester and add 2.5 mL metals-free water. Mix and continue warming for 15 minutes.
- 13.8 Remove from the block digester and cool to room temperature.
- 13.9 After cooling, dilute to 25.0 mL with metals-free water, cap tightly and shake vigorously.

#### 14.0 DATA REPORTING AND DELIVERABLES

- 14.1 All samples that require digestion and must be input as pretreated to the Laboratory Information Management System (Element™).
- 14.2 Each sample batch of up to 20 samples referencing the same digestion method is given an Element™ batch number.
- 14.2 The batch number is used to segregate digestion batches for analysis. The batch number is also used by Element™ to associate quality control with associated sample results.

#### 15.0 QUALITY ASSURANCE

- 15.1 A Digestion Blank (BLK) must be carried through the entire procedure to monitor for contamination with each digestion batch of up to 20 samples. The BLK is prepared by measuring 25 mL of metals-free water or appropriate leachate extraction fluid into a digestion cup for digestion.

Note: A dissolved metals blank is metals-free water that is matrix-matched to instrument standards at the instrument.

- 15.2 A digestion Blank Spike (BS) must be carried through the entire procedure to monitor recovery for each digestion batch.

- 15.2.1 The digestion blank spike is prepared by measuring 25.0 mL of metals-free water or appropriate leachate extraction fluid into a digestion cup and spiking with 25 µL each of two 50 mg/L mixed metals solutions (refer to Attachment 20.1).

15.2.1.1 HP2079-1-1L, stock solution A (100 mg/L) is diluted to 50 mg/L and designated QCA.

15.2.1.2 HP2079-2-1L, stock solution B (100 mg/L), is diluted to 50 mg/L and designated QCB.

- 15.2.2 A dissolved metals blank spike is prepared in metals-free water, spiked as above then matrix-matched to the instrument standards.

Approved By: <u>          gr 1-8-09          </u> QA Officer	Approved By: <u>          MAS 01-14-09          </u> Area Supervisor
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SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A SOP Number: <b>GR-01-148</b>	Revision Number: 0.3  Date Revised: 1/7/09 Date Initiated: 12/3/01
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15.3 Perform matrix spike/matrix spike duplicates (MS/MSD) for each digestion batch of up to 20 samples for each matrix type.

15.3.1 The digestion matrix spike is prepared by measuring 25.0 mL of sample into a digestion cup and spiking with 25 µL each of two 50 mg/L mixed metals solutions (refer to Attachment 20.1).

15.3.1.1 HP2079-1-1L, stock solution A (100 mg/L) is diluted to 50 mg/L and designated QCA.

15.3.1.2 HP2079-2-1L, stock solution B (100 mg/L), is diluted to 50 mg/L and designated QCB.

15.3.2 A dissolved metals matrix spike is prepared in filtered/acidified sample, matrix-matched to the instrument standards and spiked as above.

15.4 If a digestate evaporates to dryness, some metals will be volatilized. If any portion of a digestion cup bottom goes dry, discard the digestate and redigest in another batch.

15.5 Digestates must not boil during evaporation. If a digestate begins boiling, immediately remove from the block digester and reduce the temperature setting. Excessive boiling will cause analyte loss. If a digestate boils during evaporation, it must be discarded and the sample redigested in another batch.

15.6 Standard solutions must be used within the predetermined expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal and obtain or prepare another.

15.7 Quality control acceptance limits are listed in Element™.

15.8 Address unacceptable quality control and perform corrective action in accordance with TriMatrix SOP GR-10-106.

## 16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC).


16.1.1 To perform an IDC, prepare four blank spikes by spiking 25 mL of metals-free water with 25 µL of 50 mg/L spiking standard.

16.1.2 Digest as samples, following every step in the procedure and analyze.

16.1.3 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation.

16.1.4 Average recovery must fall within the blank spike control limits in Element™. Relative standard deviation must not be higher than 20%.

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Approved By:  1-8-09  
QA Officer

Approved By: KAS 01-14-09  
Area Supervisor



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SW-846 Method 3020A  
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- 16.1.5 If any criterion is not met, locate and correct the source of the problem and repeat the study successfully.
- 16.1.6 Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If multiple failures occur, correct the procedure and/or technique used, revise the procedure and repeat the study successfully.
- 16.1.7 Samples may not be processed by any analyst until an IDC study has been successfully completed.
- 16.1.8 Submit a copy of the successful IDC spreadsheet to the Quality Assurance department for training documentation.
- 16.2 Annually, a Continuing Demonstration of Capability (CDC) is required by each analyst by any of the following approaches:
- 16.2.1 By repeating the IDC study.
- 16.2.2 By inputting the last four results from a method detection limit (MDL) study to the IDC spreadsheet if prepared exclusively by the analyst.
- 16.2.3 By inputting four consecutive blank spike results obtained during the course of routine sample analysis to the IDC spreadsheet if prepared exclusively by the analyst.
- 16.2.4 By obtaining a passing result on a blind performance testing study if prepared exclusively by the analyst.
- 16.3 A method detection limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

## **17.0 POLLUTION PREVENTION**

- 17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.
- 17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.
- 17.3 Conserve the use of chemicals where applicable
- 17.4 Comply with all environmental laws associated with chemicals in the laboratory.

## **18.0 WASTE MANAGEMENT**

- 18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

Approved By:           *MS* 1-8-09            
QA Officer

Approved By:           *MAS* 01-14-09            
Area Supervisor



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Total/Dissolved Metals by ICPMS  
SW-846 Method 3020A

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
- 18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.
- 18.3 Follow all instructions in TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, for laboratory waste disposal requirements.

## 19.0 REFERENCES

- 19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition, Final Update III, Revision 1, July 1992, Method 3020A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy"*

## 20.0 ATTACHMENTS

- 20.1 Analyte List
- 20.2 Example Preparation Batch Report
- 20.3 Example Environmental Express Certificate of Analysis, Solution A
- 20.4 Example Environmental Express Certificate of Analysis, Solution B

Approved By:  1-8-09  
QA Officer

Approved By: MAS 01-14-09  
Area Supervisor



SOP Name: Block Digestion of Aqueous Samples and Extracts for Total/Dissolved Metals by ICPMS SW-846 Method 3020A	Revision Number: 0.3  Date Revised: 1/7/09 Date Initiated: 12/3/01
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**Attachment 20.1**  
**Analyte List**

Element	Environmental Express Stock Solution
Aluminum	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Antimony	HP2079-1-1L (QCB) 100 mg/L diluted to 50 mg/L
Arsenic	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Barium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Beryllium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Boron	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Cadmium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Chromium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Cobalt	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Copper	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Lead	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Manganese	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Nickel	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Selenium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Silver	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Strontium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Thallium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Tin	HP2079-1-1L (QCB) 100 mg/L diluted to 50 mg/L
Vanadium	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L
Zinc	HP2079-1-1L (QCA) 100 mg/L diluted to 50 mg/L

Approved By: <u>          <i>MS</i> 1-8-09          </u> QA Officer	Approved By: <u>          <i>MAS</i> 01-14-09          </u> Area Supervisor
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Attachment 20.2  
Example Preparation Batch Report

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0808883 Page 1 of 1

Printed: 8/11/2008 2:04:12PM

Metals, Water, 3020A Digestion

Batch comments

MAS 07-30-08 7:35AM-3:30PM  
INST=21" @9°C=19°C  
HOTBLOCK VESSEL LOT=A801LP001

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client	Source ID	Spike ID	uL Spike	Comments
0807506-01	O	Jul-30-08 07:35	MAS	25	125					
Ag Total: 6020	As Total: 6020	B Total: 6020	Ba Total: 6020	Be Total: 6020	Cd Total: 6020	Co Total: 6020	Cr Total: 6020			
Cu Total: 6020	Ni Total: 6020	Pb Total: 6020	Sb Total: 6020	Se Total: 6020	Ti Total: 6020	V Total: 6020	Zn Total: 6020			
0807506-02	E	Jul-30-08 07:35	MAS	25	125					
Ag Total: 6020	As Total: 6020	B Total: 6020	Ba Total: 6020	Be Total: 6020	Cd Total: 6020	Co Total: 6020	Cr Total: 6020			
Cu Total: 6020	Ni Total: 6020	Pb Total: 6020	Sb Total: 6020	Se Total: 6020	Ti Total: 6020	V Total: 6020	Zn Total: 6020			
0808883-BLK1		Jul-30-08 07:35	MAS	25	125					
0808883-BS1		Jul-30-08 07:35	MAS	25	125	8070952		25		
0808883-MS1		Jul-30-08 07:35	MAS	25	125	0807506-02	8070952	25		
0808883-MSD1		Jul-30-08 07:35	MAS	25	125	0807506-02	8070952	25		
0808883-PS1		Aug-04-08 09:40	DSC	2	10	0807506-02	7110622	50		

Comments	Analyst Initials
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bcb\_TM\_byAnalysis.rpt

Approved By: MA 1-8-09 Approved By: MAS 01-14-09  
QA Officer Area Supervisor



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**Attachment 20.3**  
**Example Environmental Express Certificate of Analysis, Solution A**

## ENVIRONMENTAL EXPRESS

Me 14.62.4  
 A 505048

# Certificate of Analysis

**HP2079-1-1L**  
**SOLUTION A**  
 Lot # 511212

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
High Purity Metals, Salts, or Oxides	99.99+%	HNO <sub>3</sub> , 3.5%	mg/L $\pm$ 0.5% See elements listed on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

  
 Theodore C. Rains, Ph.D.  
 Laboratory Director

Exp. Date RAY 06  
**MSDS ATTACHED**

490 Wando Park Blvd.  
 Mt. Pleasant, South Carolina 29464  
 Phone: 1.843.881.6560  
 Toll Free: 1.800.343.5319  
 FAX: 1.843.881.3964  
[www.environmentalexpress.com](http://www.environmentalexpress.com)



Approved By:   
 QA Officer

Approved By: MAS 01-14-09  
 Area Supervisor



SOP Name: Block Digestion of Aqueous Samples and Extracts for  
Total/Dissolved Metals by ICPMS  
SW-846 Method 3020A  
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Attachment 20.4  
Example Environmental Express Certificate of Analysis, Solution B

## ENVIRONMENTAL EXPRESS

# Certificate of Analysis

**HP2079-1-1L**  
**Solution B**  
Lot # 631157

6110557  
02  
11-15-06  
EXP. 12-01-07

<u>Source</u>	<u>Source Purity</u>	<u>Matrix</u>	<u>Standard Concentration</u>
High Purity Metals, Salts or Oxides	99.98+%	HNO <sub>3</sub> , 2% + Tr IIF	100 mg/L ± 0.5% Antimony Silicon from (NH <sub>4</sub> ) <sub>2</sub> SiF <sub>6</sub> Titanium Molybdenum Tin

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.


  
Theodore C. Rains, Ph.D.  
Laboratory Director

Exp Date: DEC 07

**MSDS ATTACHED**

490 Wando Park Blvd.  
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FAX: 1.843.881.3964  
www.environmentalexpress.com



Approved By:  1-8-09  
QA Officer

Approved By: MAS 01-14-09  
Area Supervisor





## STANDARD OPERATING PROCEDURE

Base/Neutral/Acid Compounds by  
Gas Chromatography/Mass Spectrometry

EPA Method 625  
SW-846 Method 8270C

### APPROVALS:

Operations Manager: Jeff P. Glaser Date: 1/23/09  
QA Officer: Tom C. Boocher Date: 1-22-09  
Area Supervisor: Janet M. Kudirka Date: 1/23/09

Procedure Number: GR-04-103  
Revision Number: 5.5

Date Initiated: 12/9/98  
Effective Date: 2/20/09

Date Revised: 1/22/09  
Pages Revised: All

By: Janet M. Kudirka  
Total Number of Pages: 46

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>4-16-10</u>	<u>Godi ZBlum</u>	<u>4-16-11</u>
_____	_____	_____
_____	_____	_____



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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
EPA Method 625, SW-846 Method 8270C	Date Revised: 1/22/09
SOP Number: <b>GR-04-103</b>	Date Initiated: 12/9/98

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## 1.0 SCOPE AND APPLICATION

- 1.1 This procedure is used to determine the concentration of semi-volatile organic compounds in extracts of various solid waste matrices, soils, groundwaters, and wastewaters.
- 1.2 This procedure is used to quantify most basic, neutral, and acid extractable organics that are soluble in methylene chloride, and capable of elution without derivatization as sharp peaks from a gas chromatograph, fused-silica capillary column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. Refer to Table 1 for routine compounds, retention times, primary and secondary quantitation ions, internal standards, and surrogates. Table 1A lists additional analytes that are amenable to this procedure.
- 1.3 The following compounds may require special treatment. Benzidine can be subject to oxidative loss during solvent concentration. The chromatography must be monitored for peak broadening and tailing. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction with acetone, and photochemical decomposition. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. 1,2-diphenylhydrazine also decomposes in the injection port into azobenzene. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling materials.
- 1.4 Default reporting limits for clean extracted samples using 1000 mL or 30 g concentrated to 10 mL and analyzed by this procedure are listed in Tables 2 and 2A. Reporting limits will be proportionately higher depending upon the sample matrix, the preparation technique and extract dilutions. If a client requires lower reporting limits than those in Tables 2 and 2A, the final extract volume may be concentrated to 1.0 mL.
- 1.5 This procedure is restricted to use by or under the supervision of analysts experienced in gas chromatography/mass spectrometry, and skilled in mass spectra interpretation. Each analyst must demonstrate the ability to generate acceptable results.

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996*
- 2.2 *40 Code of Federal Regulations, most current edition, Pt. 136, App. A, Method 625-Base/Neutrals and Acids*
- 2.3 *40 Code of Federal Regulations, most current edition, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit*

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Approved By: <u>DM 1-22-09</u>	Approved By: <u>JAC 1/23/19</u>
QA Officer	Area Supervisor



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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
EPA Method 625, SW-846 Method 8270C	Date Revised: 1/22/09
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### 3.0 SUMMARY OF PROCEDURE

- 3.1 Before analysis, samples must be extracted using appropriate sample preparation and cleanup techniques. Waters will be extracted using TriMatrix SOP GR-09-101. Soils will be extracted using TriMatrix SOP GR-09-103. Extracts are analyzed by injection into a gas chromatograph, with mass spectrometric identification and detection of compounds. Quantitation is made by comparison with standards.
- 3.2 Wastewater sample analysis for NPDES permits under the Clean Water Act follows USEPA Method 625, requiring the following modifications to this procedure.
- 3.2.1 DFTPP tunes and continuing calibration standards are analyzed once every 24 hours instead of every 12.
- 3.2.2 A 3-point calibration may be used in place of a 6-point calibration. Standard practice, however will generally be to quantify using the 6-point calibration. System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) are not used. Instead the response factors (RF) for every compound of interest must have <35% RSD to use the average RF for quantitation. If these criteria are not met, a new calibration curve must be constructed.
- 3.2.3 The continuing calibration standard does not use SPCCs and CCCs. The RF for compounds of interest in the continuing calibration standard that is on the 625 list must be  $\leq 20\%$  difference when compared against the calibration. If not, a new continuing calibration standard will be run with acceptable percent differences, or a new 3-point or 6-point calibration will be run.
- 3.2.4 There are no criteria for internal standard areas or retention times in Method 625. Method 8270C criteria will be followed.
- 3.2.5 Every compound of interest must be in matrix spikes and Laboratory Fortified Blanks, not just the limited list specified in Method 8270C. Laboratory generated windows for surrogate and spike recoveries are calculated as specified in this procedure.

### 4.0 PARAMETER OR COMPOUND LIST

- 4.1 Analytes for analysis by Method 8270C or Method 625 are listed in Table 1 and 1A. Other analytes may be determined providing a demonstration of capability study is successfully completed. In addition, QC criteria described for Table 1 and 1A compounds must be considered the minimum acceptable for additional analytes.

### 5.0 REFERENCED SOPs

- 5.1 TriMatrix Laboratories SOP GR-09-101, *The Extraction of BNAs in Water*, latest revision
- 5.2 TriMatrix Laboratories SOP GR-09-103, *Extraction of Semi-Volatile BNAs in Soil, Sediment and Sludge*, latest revision

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Approved By: <u>          JD 1-22-09          </u>	Approved By: <u>          JMK 1/23/19          </u>
QA Officer	Area Supervisor



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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry EPA Method 625, SW-846 Method 8270C	Revision Number: 5.5
SOP Number: <b>GR-04-103</b>	Date Revised: 1/22/09
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- 5.3 TriMatrix Laboratories SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision
- 5.4 TriMatrix Laboratories SOP GR-09-104, *Waste Dilution of BNAs*, latest revision
- 5.5 TriMatrix Laboratories SOP GR-15-102, *Waste Disposal*, latest revision
- 5.6 TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision
- 5.7 TriMatrix Laboratories SOP GR-10-123, *ELEMENT Data Transfer and Reviews*, latest revision

## 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

- 6.1 Interferences can be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware, leading to discrete artifacts and/or elevated baselines in the total ion current profiles. Do not use plastic materials, including polypropylene squirt bottles. Phthalate esters leaching from plastics can result in false positives. All materials used in extraction and analysis must routinely be demonstrated to be interference-free by running method performance blanks. Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use. Use only high purity reagents (reagent grade or better) and solvents (pesticide grade or better). Solvents used for extraction and analysis must be tested prior to use. Once they are proven to be contaminant-free, large lots are sequestered from the vendor to ensure purity.
- 6.2 Carryover can occur whenever highly concentrated samples precede the analysis of low or non-detect samples. To reduce carryover, autosampler syringes are rinsed between extract injections. Whenever possible, the analyst should analyze a reagent blank immediately after a high level sample. If carryover is suspected, all affected samples must be re-analyzed.
- 6.3 Corrective actions for interference and contamination are outlined in TriMatrix SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision.

## 7.0 SAFETY PRECAUTIONS

- 7.1 Analysts must comply with all instructions for health and safety as outlined in the Laboratory Safety Manual and Chemical Hygiene Plan. Safety glasses are mandatory in all laboratory areas, except when entering data at computer terminals.
- 7.2 Methylene chloride and most target analytes, surrogates, and spikes are highly toxic and suspected carcinogens. Every precaution must be taken to avoid dermal contact, ingestion and inhalation. Wear appropriate protective clothing and disposable gloves when handling any of the above compounds. Prepare standards and extracts for analysis under a fume hood.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

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Approved By: <u>          Jm 1-22-09          </u>	Approved By: <u>          Jm 1/23/11          </u>
QA Officer	Area Supervisor



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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
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- 8.1 To achieve the reporting limits in Tables 2 and 2A, a minimum 30 g of soil or 1000 mL of aqueous sample must be collected and extracted. Additional sample volume is necessary if matrix spikes are required.
- 8.2 Samples should be collected in amber glass jars, with PTFE-lined screw-cap lids. These jars must meet or exceed current USEPA cleanliness standards. Plastic containers and/or plastic-lined lids must NOT be used due to potential phthalate ester contamination.
- 8.3 Samples must be stored away from light, at 4° C ± 2° C. Store extracts away from light and ≤ -10° C.
- 8.4 Analysis must be performed within 40 days of sample extraction.

## 9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

- 9.1 Gas chromatograph/mass spectrometer system – this procedure is applicable to either of the systems described in 9.1.1 or 9.1.2.

9.1.1 Varian Saturn II ion trap mass spectrometer is set up to scan from 40-450 mass units twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The spectrometer produces a mass spectrum that meets all criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

9.1.1.1 Ion Trap operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-450 amu
- Scan time: 820 amu/second = 2 scan/second
- Source temperature: 190° C
- Transfer line temperature: 250° C

9.1.1.2 Varian 3400 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C until benzo[g,h,i]perylene has eluted.
- SPI Injector Temperature Program: 45° C for 0.5 minute, then to 300° C at 100°C/minute.
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 6 psi (~ 1 to 2 mL/minute)

9.1.2 The Agilent 5973 Mass Selective Detector (MSD) is set up to scan from 40-550 mass units at least twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The detector produces a mass spectrum that meets all the criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

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9.1.2.1 MS operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-550 amu
- Scan time: sampling rate of 3 = 2.89 scan/second
- Source temperature: 230° C
- Transfer line temperature: 280° C

9.1.2.2 Agilent 6890 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C for 4.0 minutes
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 1.0 mL/minute in constant flow mode

9.1.3 Injector

9.1.3.1 A Septum Programmable Injector (SPI), temperature programmed. The injector is capable of sub-ambient initial injector temperatures, followed by rapid heating up to 315° C, and is used for Ion Trap applications.

9.1.3.2 A Split/splitless injector in the splitless mode held at 250° C, is used for the Mass Selective Detector (MSD).

9.1.4 Fused Silica Capillary Columns

9.1.4.1 A 30 m x 0.32mm i.d. 1.0 um film thickness silicon coated DB-5MS or equivalent, is used for the Ion Trap.

9.1.4.2 A 30 m x 0.25 mm, i.d. 0.25 um film thickness silicon coated DB-5MS or equivalent, is used for the Mass Selective Detector (MSD).

9.1.4.3 A 20 m x 0.15 mm. i.d. 0.15 um film thickness silicon coated factorFour VF-5MS is also used for the Mass Selective Detector (MSD).

9.1.5 Data system:

9.1.5.1 A multi-tasking personal computer, capable of the continuous acquisition and storage on magnetic tape or compact disk, of all mass spectra obtained throughout a chromatographic program.

9.1.5.2 The software can search acquired GC/MS data files, for specific mass ions, and can plot ion abundances versus time or scan number. This type of plot is an Extracted

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Ion Current Profile (EICP). The software integrates abundances in any EICP between specified time or scan number limits. The 1992 (Ion Trap) or 1998 (MSD) version of the NIST mass spectral library is referenced for identification of unknowns.

- 9.1.6 Autosampler: A Leap A200SE or equivalent, programmed for 1.0 uL injections with multiple rinses before and after injection.

## **10.0 ROUTINE PREVENTIVE MAINTENANCE**

- 10.1 When chromatography degrades or sensitivity decreases, routine maintenance consists of cleaning/changing the injection port liner, clipping capillary column, changing injection port septum, and cleaning the source.
- 10.2 The rough pump oils must be replaced at least annually.
- 10.3 Check the MSD diffusion pump oil every six months and refill, if necessary.
- 10.4 Check the Ion Trap turbo pump wick annually and re-oil or replace, if necessary.
- 10.5 If column performance deteriorates, performance may be brought back by repeated injections of Toluene overnight. If this is ineffective, the column will need replaced.

## **11.0 CHEMICALS AND REAGENTS**

- 11.1 All chemicals and reagents used must be reagent grade or better, and must be determined free of interferences.
- 11.2 Acetone, hexane, methylene chloride, and toluene must be pesticide grade or better, and must be determined free of interferences.

## **12.0 STANDARDS PREPARATION**

- 12.1 Stock calibration standard solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions.
- 12.1.1 Commercially purchased stock solutions are used to prepare the majority of working standards. The purchased concentration for stock mixes is typically 2000 mg/L. Solutions arrive at the laboratory sealed in various glass ampules, which are combined during preparation.
- 12.1.1.1 Stock standards are combined with the surrogate currently being used during sample extraction, to create a 100 mg/L calibration standard in methylene chloride.
- 12.1.1.2 Diluting from the 100 mg/L standard, make up the 8-point initial calibration at concentrations of 25, 20, 15, 10, 5.0, 2.0, 1.0, and 0.5 mg/L. Due to low response

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in the lowest calibration point, prepare extra concentrations of 2,4-dinitrophenol, 4-nitrophenol, benzoic acid and benzidine for a final concentration of 1.0 mg/L to 50 mg/L.

- 12.1.2 Stock standard solutions from neat compounds are prepared by weighing out each analyte to the nearest tenth of a milligram. The material is dissolved in methylene chloride or other suitable solvent and diluted to volume in a volumetric flask. If compound purity is 96% or greater, the weighed mass is used without correction to calculate stock standard concentration.
- 12.1.3 Transfer stock standard solutions to screw-cap vials with PTFE-lined lids and store below -5° C, protected from light. Stock standard solutions must be monitored for degradation or evaporation.
- 12.1.4 All stock standard mixes must be replaced annually or sooner if comparison with quality control checks indicates a problem. Working calibration standards must be replaced every six months or sooner if comparison with quality control checks indicates a problem. Continuing calibration standards must be prepared weekly.
- 12.2 Internal standard solutions: The six internal standards used are 1,4-dichlorobenzene-d<sub>4</sub>, acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub>, naphthalene-d<sub>8</sub>, and perylene-d<sub>12</sub>. The stock standard is prepared in a volumetric flask at 1000 ug/mL by dissolving 100 mg of each compound with a small volume of methylene chloride then diluting to 100 mL. Each 1.0 mL extract analyzed must be spiked with 5.0 uL of the internal standard solution. The resulting concentration of internal standards in extracts is 5.0 ug/mL.
- 12.3 GC/MS tuning standard: The standard is used for tuning the mass spectrometers and contains 5.0 ng/uL of decafluorotriphenylphosphine (DFTPP), 50 ng/uL of pentachlorophenol, 50 ng/uL of benzidine, and 50 ng/uL of 4,4'-DDT, in methylene chloride. Stock standards for the tuning mixture are purchased from a vendor at 1000 mg/L. Appropriate dilutions for the working concentrations are prepared from these standards. The tuning standard also verifies injection port inertness and monitors GC column performance.
- 12.4 Surrogate standards: Surrogate solution contains o-terphenyl, 2-fluorophenol, phenol-d<sub>6</sub>, 2,4,6-tribromophenol, nitrobenzene-d<sub>5</sub>, and 2-fluorobiphenyl. These are made as follows.
- 12.4.1 Acid and base compound surrogate solutions are made separately from neat materials:
- 12.4.1.1 Add 0.5 g of each base-neutral surrogate to a 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.
- 12.4.1.2 Add 1.0 g of each acid surrogate to another 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.
- 12.4.1.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. The finished solution must be checked for accuracy by analyzing on the GC/MS. Once approved, the prep lab and the

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mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.

12.4.1.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the organic prep laboratory refrigerator at  $4 \pm 2^\circ \text{C}$ .

Surrogate	Mass (g)	Dilution Volume (mL)	Stock Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d <sub>5</sub>	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d <sub>6</sub>	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

12.4.2 The volume spiked is 100 uL for each 1.0 mL of extract volume, resulting in a concentration of 10 ng/uL of base/neutral surrogates and 20 ng/uL acids.

12.5 Spiking standards applicable to Laboratory Blank Spikes (BS) and Matrix Spikes (MS/MSD): The base/neutral spike solution contains acenaphthene, 1,4-dichlorobenzene, 2,4-dinitrotoluene, naphthalene, N-nitrosodi-n-propylamine, pyrene, and 1,2,4-trichlorobenzene. The acid spike contains 4-chloro-3-methyl phenol, 2-chlorophenol, 4-nitrophenol, pentachlorophenol, and phenol. The spikes are made up in methanol at 100 ug/mL for the base/neutrals and 200 ug/mL for the acids, as detailed in the appropriate extraction procedure. The volume spiked is 100 uL per 1.0 mL of extract, resulting in a concentration of 10 ng/uL for base/neutrals, and 20 ng/uL for acids.

**NOTE: For all State of Wisconsin samples and when other projects require it, every analyte of interest must be spiked into the BS and MS/MSDs or only the BS, as applicable. All analytes must be spiked at 10 ng/uL before extraction.**

### 13.0 SAMPLE PREPARATION

13.1 For sample extractions, refer to the appropriate procedures in section 5.0.

13.2 All samples must be spiked with surrogates before extracting (Refer to section 12.4).

13.3 All extracts must be spiked with internal standards, immediately prior to injection (Refer to section 12.2).

13.4 Extracts that are dark in color or thick and viscous will be diluted at least 1:10 prior to analysis.

### 14.0 CALIBRATION PROCEDURES

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- 14.1 Set up instrumentation as specified in section 9.1.1 and 9.1.2.
- 14.2 All samples and standards are introduced into the GC/MS, by 1.0 uL direct injection. All injections are performed using an autosampler.
- 14.3 Each GC/MS system is hardware-tuned to meet Table 3 or Table 3A criteria for a 5.0 ng, 1.0 uL DFTPP injection. No analysis may begin until these criteria are met. Mass spectra are taken across the 198 ion apex. Background subtraction is designed only to eliminate column bleed or instrument background ions.
- 14.3.1 The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and/or DDD must not exceed 20%. Calculate degradation as follows:
- $$\% \text{ degradation} = \frac{\text{Area}_{\text{DDE}} + \text{Area}_{\text{DDD}}}{\text{Area}_{\text{DDE}} + \text{Area}_{\text{DDD}} + \text{Area}_{\text{DDT}}} \times 100$$
- 14.3.2 The degradation result must be recorded in the daily instrument run logbook.
- 14.3.3 Pentachlorophenol and benzidine must be present at their normal responses, with minimal peak tailing. If peak tailing is suspect, calculate a tailing factor using the pentachlorophenol total ion chromatogram. To determine if corrective action is required, follow Figure 1 instructions. A tailing factor of 1 to 2 is considered acceptable, a tailing factor of 2 to 4 is considered the warning limit, and a tailing factor of 4 or higher is considered unacceptable, requiring corrective action. Corrective action for degradation and/or poor chromatography can include cleaning the injection port and clipping the first 6 to 12 cm off the capillary column.
- 14.4 All analytes must be quantified using the method of internal standards. Analytes and their corresponding internal standards are provided in Tables 1 and 1A. For analytes not listed, the corresponding internal standard must be the one eluting nearest the unlisted analyte. Primary quantitation ions are also provided in Tables 1 and 1A. A secondary ion may be used for quantitation, only if interferences are noted. If a secondary ion is used, the compound RF must be recalculated before quantitation.
- 14.5 Initial Calibration:
- 14.5.1 Calibration standards: The initial calibration for all analytes of interest must contain a minimum of five points (three for method 625). Table 1 standards calibration is typically run at 0.5, 1.0, 2.0, 5.0, 10, 15, 20, 25 ug/mL. The 25 ug/mL standard may be omitted. However, omitting lowers the linear calibration range. Each 1.0 mL calibration standard aliquot must be spiked with 5.0 uL of internal standard solution, immediately prior to analysis. Table 1A and other analytes, are run at the appropriate concentration, based on response. The lowest concentration of the initial calibration must be at the reporting limit.
- 14.5.2 Injections of 1.0 uL for each calibration standard, including surrogates and internal standards are analyzed. Figures 2 and 2A show chromatograms of a continuing calibration standard. A Response Factor (RF) is calculated for each compound using the Table 1 or 1A primary ions. Response factors are calculated as follows:

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$$RF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

where:

$A_X$  = Area of the primary ion for the compound being measured  
 $A_{IS}$  = Area of the primary ion for the specific internal standard  
 $C_X$  = Concentration of the compound being measured (ng/uL)  
 $C_{IS}$  = Concentration of the specific internal standard (ng/uL)

14.5.3 A system performance check must be performed to ensure that average RFs are acceptable for use in quantitation. System Performance Check Compounds (SPCCs) are listed in Table 4. The minimum acceptable average RF for SPCC compounds is 0.050. SPCCs typically have very low RFs (0.1 to 0.2) and tend to decrease in response as the chromatographic system or the standards deteriorate. SPCCs are usually the first to show poor performance, and must meet the minimum requirement when the system is calibrated. The minimum recommended RF for all non-SPCC analytes of interest is 0.01, however, spectra quality and background (signal to noise) must also be considered.

14.5.4 Percent relative standard deviation (RSD) must be calculated for each compound using the following equation:

$$\%RSD = \frac{SD}{RF} \times 100$$

where:

SD = The standard deviation of the average response factor across the initial calibration curve.  
 RF = Average response factor across the initial calibration curve.

14.5.5 Percent RSD for each compound must be less than 15% to use an RF for quantitation, which assumes linearity through the origin. Percent RSD for each Table 5 calibration check compound (CCC) **must** be less than 30%. If percent RSD for any CCC is between 15% and 30%, a calibration curve must be constructed as outlined in 14.5.8. If percent RSD is **greater** than 30% for any CCC, the system is too unstable to proceed. Corrective action, including replacing the injection port liner, replacing or clipping the capillary column, and recalibration of the GC/MS system, must be initiated.

14.5.6 The relative retention time of each compound of interest in each calibration run must agree within 0.06 relative retention time units.

14.5.7 If percent RSD of any non-CCC compound is 15% or less, the response factor is assumed to be constant over the calibration range, and the average response factor can be used for quantitation.

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14.5.8 If percent RSD for any compound is greater than 15%, a regression curve must be constructed by plotting area ratio ( $\text{Area}_{\text{analyte}}/\text{Area}_{\text{IS}}$ ) against concentration. A first order or higher regression fit is used. The curve must not be forced through the origin:

14.5.8.1 Linear regression requires five calibration standards and is calculated as follows:

$$C_s = [(A_s \cdot C_{is}) / A_{is}] - b / a$$

Where:

$A_s$  = Area of the target analyte peak, in sample "s"

$A_{is}$  = Area of the "is" internal standard peak

$C_s$  = Concentration of the target analyte in calibration standard "s"

$C_{is}$  = Concentration of the internal standard "is"

a = Slope of the curve (also called the coefficient of C)

b = The intercept

14.5.8.2 A quadratic (second order) fit requires six calibration standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s \cdot C_{is}) / A_{is}] = a \cdot C_s^2 + b \cdot C_s + c$$

Where:

c = the constant

14.5.8.3 A third order polynomial requires seven standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s \cdot C_{is}) / A_{is}] = a \cdot C_s^3 + b \cdot C_s^2 + c \cdot C_s + d$$

Where:

d = the constant

14.5.8.4 Analysts may select each analyte regression curve giving the least calibration error, as indicated by the correlation coefficient. To be acceptable, the correlation coefficient "r" for a first order regression must be 0.99 or higher. For second and third order regressions, the coefficient of determination (COD) "r<sup>2</sup>" must be 0.99 or higher.

NOTE: Non-linear regression calibration must not be used as compensation for improper instrument maintenance. Non-linear regression for any analyte must not be employed on an instrument previously shown to **routinely** exhibit linearity.

14.5.9 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

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- 14.5.9.1 A minimum of six calibration points are required to use second order regression curves. Five data points are needed for first order.
- 14.5.9.2 The lowest calibration point defines an analyte reporting limit. The low point can only be dropped provided required reporting limits are not affected.
- 14.5.9.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.
- 14.5.10 Run a second-source calibration standard containing all analytes at a mid-range concentration following the initial calibration. All analytes must have recoveries between 75-125% for the initial calibration to be considered valid. If these criteria are not met, analyze the second-source standard a second time. If the second analysis fails, take corrective action up to and including preparation and running of new initial calibration standard solutions. Repeat the second-source analysis for confirmation that corrective action was effective.
- 14.6 Continuing Calibration:
- 14.6.1 Prior to analysis of samples or standards, the DFTPP tuning standard is analyzed and evaluated, as specified in section 14.3. Acceptable DFTPP tuning must be demonstrated at the beginning of each 12-hour shift.
- 14.6.2 Immediately following every acceptable DFTPP analysis, a continuing calibration standard must be run at a mid-level concentration. For method 8270C analytes listed in Table 1, the concentration is 10 ug/mL. A continuing calibration standard contains all semi-volatile target analytes, including all surrogates and internal standards. Table 1A and other analytes, are run at the appropriate concentration, based on response. Each analyte RF is compared with the initial calibration average RF, from the same instrument.
- 14.6.3 System Performance Check Compounds (SPCC): A system performance check is made on every continuing calibration standard. All SPCC criteria must be met for a continuing calibration standard to pass. Table 4 lists SPCC compounds used for the continuing calibration. The minimum SPCC RF is 0.050. The recommended minimum RF for all non-SPCC compounds of interest is 0.01. However, spectra quality and background (signal to noise) must also be considered. If minimum response factors are not met for SPCCs, the system must be evaluated and corrective action taken, before analysis can continue. Possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front of the analytical column, and active sites in the column or chromatographic system.
- 14.6.4 Calibration Check Compounds (CCC): After the system performance check is met, Table 5 CCCs are used to check the initial calibration validity.

When average response factor is used for calibration, calculate percent difference between the initial calibration average RF and the continuing calibration RF:

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$$\% \text{ Difference} = \frac{RF_v - RF_m}{RF_m} \times 100$$

where:

RF<sub>v</sub> = response factor from verification standard

RF<sub>m</sub> = average calibration factor from initial calibration

Use percent drift to determine acceptance when calibrating using a regression fit model:

$$\% \text{ Drift} = \frac{C_c - C_1}{C_1} \times 100$$

where:

C<sub>1</sub> = theoretical CCC standard concentration

C<sub>c</sub> = measured concentration using appropriate regression fit.

14.6.5 If percent difference or drift for every CCC is less than  $\pm 20\%$ , the initial calibration remains valid. If any CCC compound differs from the initial calibration curve by greater than  $\pm 20\%$ , corrective action must be taken. Problems similar to those listed under section 14.6.3 can also affect CCC results. If the problem source cannot be determined after corrective action has taken place, the calibration is considered out of control. At a minimum, the continuing calibration standard must be re-run. If necessary, a new calibration will be run. All CCC criteria must be met before sample analysis can continue. For non-CCC analytes, the percent difference or drift must be  $\leq 25\%$ , with the exception of poor performing analytes, which can have up to four outside of  $\leq 25\%$ , but must be  $\leq 40\%$ . Examples of poor performing analytes include: Chloro- and Nitro-anilines, Benzoic acid, Benzidine, Hexachlorocyclopentadiene, 4-Nitrophenol, and 2,4-Dinitrophenol. In addition, if response for any non-CCC compound is outside this criteria, but is recovering on the high side (response for the analyte is out of control high), then extracts with non-detect results for these compounds may still be used since there is no question of analyte sensitivity for the day. For method 625, all compounds must have a percent difference of less than  $\leq 20\%$ .

14.6.6 Internal standard responses and retention times in continuing calibration verification standards and samples must be evaluated in each run. If retention time for any internal standard varies by more than 30 seconds from the last calibration verification, the system must be inspected for malfunctions, and corrective action performed as necessary. If the EICP area for any internal standard varies by a factor of 2 (-50% to +100%) from the midpoint standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrective action performed as necessary. All sample extracts analyzed under either of these out of control conditions must be reanalyzed.

## 15.0 ANALYTICAL PROCEDURE

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- 15.1 Every 12-hour shift **must** begin with the analysis of DFTPP, followed by a new calibration or a continuing calibration verification standard, then followed by an instrument blank.
- 15.2 The DFTPP tune **must** pass the tuning criteria in section 14.3 before subsequent analysis can begin. The 12-hour acquisition window **must** begin at the injection time of DFTPP. The entire 12-hour shift must be run under the same mass spectrometer conditions used for DFTPP. All samples must be run under the same gas chromatographic conditions as the initial calibration. Refer to sections 9.1.1 and 9.1.2 for recommended operating conditions.
- 15.3 Any calibrated compound **must** have an acceptable average Response Factor (RF) generated from the initial calibration curve, or be calibrated using a first or higher order regression curve. If Method 8270C is being utilized, the calibration **must** pass the SPCC and CCC criteria specified in section 14.5.
- 15.4 A continuing calibration standard is run on days an initial calibration is not. The continuing calibration standard **must** pass the criteria specified in section 14.6.
- 15.4.1 After the DFTPP and continuing calibration criteria are met, the analytical system **must** be demonstrated free from contamination by running an instrument blank before sample analysis during each 12-hour shift (Refer to section 18.3).
- 15.5 Once the above criteria are met, sample analysis may proceed for the remainder of the 12-hour shift.
- 15.6 If response for any analyte in the sample exceeds the initial calibration range, a dilution of the extract must be made. Attempt to dilute the sample only enough to bring the analyte(s) in question into the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the required 5.0 ng/uL internal standard concentration. The diluted extract is then reanalyzed.
- 15.7 A matrix spike, matrix spike duplicate, method preparation blank, and laboratory fortified blank **must** be run for each quality control batch to assess extraction efficiency. Up to a maximum of 20 samples/batch is allowed. It is preferred but not required that matrix quality control be analyzed with samples from the same extraction batch during one 12-hour shift. Additional quality control requirements are specified in section 18.0.

## 16.0 CALCULATIONS AND DATA HANDLING

### 16.1 Qualitative analysis:

16.1.1 An analyte is identified by a mass spectra comparison between the sample and a standard of the suspected compound (standard reference spectrum). Standard reference spectra are obtained through analysis of the calibration. Two criteria must be satisfied to verify identification. First, a sample component must elute at the same GC relative retention time (RRT) as the standard compound. Secondly, the mass spectra of the sample component and standard compound must agree.

16.1.1.1 The sample component RRT must fall within  $\pm 0.06$  RRT units of the standard compound, in the daily continuing calibration standard. If interfering co-elution

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prohibits accurate sample component RRT measurement from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles unique to the compound of interest.

16.1.1.2 All ions in standard mass spectra at a relative intensity greater than 10% are automatically checked by the software to be in a sample spectrum. The most abundant ion in standard mass spectra equals 100%. Relative ion intensities between standard and sample spectra, must agree within  $\pm 30\%$ . For example, an ion with 50% abundance in standard spectra, means the corresponding sample abundance must be between 20% and 80%.

16.1.2 For samples containing components not associated with calibration standards, a library search can be made for a tentative identification. Only after visual comparison of sample spectra with the nearest library searches must the analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description must be given to identify functional groups. TIC nomenclature by functional group is as follows:

Name	CAS #
Unknown Alcohol	XX-XX-XX
Unknown Freon	XX-XXX-XXXX
Unknown Ketone	XX-XXX-XX
Unknown Acid	XXX-XX-XXXX
Unknown Hydrocarbon	XXX-XXX
Unknown Glycol Ethers	XXX-XX-X
Unknown Substituted Benzenes	XXXXX
Hydrocarbons (sub. Benzenes)	XXXXX-X-X
Hydrocarbons, Total	XXXXXX

Guidelines for making tentative identification are:

- 16.1.2.1 Relative intensities of major ions in a reference spectrum (ions with  $>10\%$  of the most abundant ion) should be present in the sample spectrum.
- 16.1.2.2 Relative intensities of major ions should agree within  $\pm 20\%$ . For example, an ion with 50% abundance in a standard spectrum corresponds to a sample ion abundance between 30% and 70%.
- 16.1.2.3 Molecular ions in the reference spectrum should be present in the sample spectrum.
- 16.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or co-eluting components.
- 16.1.2.5 Ions in the reference spectrum but not in the sample spectrum should be reviewed for subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create such discrepancies. TIC peaks are determined by the following search criteria:

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16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 20 potential TICs must be reviewed, unless otherwise required. The match quality must be 70% or higher to report a positive identification, in the absence of interfering peaks.

NOTE: A 70% match corresponds to a fit of 700 on the Ion Trap or a Q-value of 70 on the MSD. If there are interfering peaks, analyst discretion must be employed when reporting matches.

## 16.2 Quantitative analysis:

16.2.1 When a compound has been identified, quantitation is performed using area of the appropriate quantitation ion(s), listed in Tables 1 and 1A. The internal standards technique is used. Internal standards are specified in Tables 1 and 1A. For target analytes in neither table, the internal standard nearest the analyte retention time of the analyte must be used. All qualitative results must be reported without correction for recovery, and without blank subtraction.

16.2.2 Calculate analyte concentration as follows:

### Water:

$$\text{Concentration in ug / L} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(V_o)}$$

where:

$A_x$  = Characteristic ion area, for the compound being quantitated  
 $I_s$  = Internal standard concentration injected, in ug/mL  
 $V_t$  = Total extract volume, in mL  
 $A_{is}$  = Internal standard characteristic ion area  
 $\text{RF}$  = Average response factor, for compound being quantitated (section 14.5.2)  
 $V_o$  = Sample volume extracted, in liters  
 $\text{Dil}$  = Any post extraction dilution factor

### Sediment/Soil Sludge:

$$\text{Concentration in mg / kg} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(W_s)(D)}$$

where:

$W_s$  = Sample mass extracted, in grams  
 $D$  = Sample % solids, as a decimal fraction

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All other variables are the same..

NOTE: If any analyte initial calibration RF has not met the 15% RSD requirement, a regression curve must be used to determine extract concentration and the calculations modified accordingly.

16.2.3 When requested, the concentration of non-calibrated sample components must be estimated. The formulas above are used with the following modifications:

16.2.3.1 Areas  $A_x$  and  $A_{is}$  are from total ion chromatograms, and the compound RF is assumed to be 1.

16.2.3.2 Use the nearest interference-free internal standard.

16.2.3.3 The concentration reported must be qualified as estimated.

### 16.3 Manual Integrations

16.3.1 The TriMatrix policy concerning manual integration must be followed without exception. Every effort must be made to set up data processing software integration parameters, to avoid manual integration.

16.3.2 All manual integration must be documented by printing both the original integration performed by the data system, and the manually integrated ion chromatograms. Manual integrations must be initialed and dated, and must have the rationale for the new integration clearly stated on the print out. These must be included in all client package deliverables.

16.3.3 When required, manual integration must be consistently applied to standards and samples. Examples where manual integration may be necessary include: closely eluting isomers (1,3- and 1,4-Dichlorobenzene), jagged analyte peak shapes split by the integrator, and analytes like benzidine that have significant peak-tailing.

16.3.4 **Under no circumstances must manual integration be applied solely for the purpose of meeting quality control. Peak shaving and peak enhancing are strictly prohibited.**

16.3.5 For further details, refer to TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision.

## 17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are also responsible for correctly filling in, turning in, and filing the associated paperwork. It is essential to perform these tasks, to provide defensible data to clients.

### 17.2 ELEMENT Reporting

17.2.1 When an analysis sequence is completed, all data must be uploaded to the ELEMENT DataSystem. For details on how to upload data, refer to TriMatrix SOP GR-10-123. Carefully

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review the *data entry* table to insure that results are accurately reported and associated with the correct quality control batch. Manually enter a dilution factor for each corresponding sample. Dilution factors are used to calculate elevated reporting limits from diluting an extract. The dilution factor is a multiplier that may or may not reflect actual dilution volumes.

- 17.2.2 If internal chain-of-custody is required, it is important that a COC form be filled in correctly, and returned to the COC file location.

### 17.3 Laboratory Required Paperwork

- 17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a write-over, and must be dated and initialized. Blank lines in run logbooks must be Z'd out. Refer to Figures 3 and 4 for standards forms and run logbook examples.
- 17.3.2 All tune, calibration, and continuing calibration standard runs must be placed in the correct file.
- 17.3.3 All ELEMENT documentation and raw data must be placed in the correct client folder and given to a data management technician. The technician must record the date and contents of the data then archive the folder.

### 17.4 CLP-like Deliverables

- 17.4.1 Include the following in data packages requiring CLP-like deliverables:
- 17.4.1.1 Copies of tunes, including the EICP. These copies must contain ion peaks for DFTPP, Benzidine, and Pentachlorophenol with tailing factor calculations and results, ion abundance results and requirements including mass listings, and associated Form Vs.
  - 17.4.1.2 Copies of calibrations, including raw data quantitation reports, chromatograms, and associated Form VIs.
  - 17.4.1.3 Copies of continuing calibration standards, including raw data quantitation reports, chromatograms, and associated Form VIIIs.
  - 17.4.1.4 Copies of raw sample data, including applicable quality control, quantitation reports, triple plots for positive results, chromatograms, and TIC reports when requested
  - 17.4.1.5 Completed MPB summary Form IVs.
  - 17.4.1.6 Completed Internal Standard recovery Form VIIIs.
  - 17.4.1.7 Copies of supporting information, including: internal chain of custody forms, run and standards logbooks, method detection limits, and extraction summaries.

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NOTE: Forms I, II, and III are generated from ELEMENT and must be included with the deliverables package.

## 18.0 QUALITY ASSURANCE

18.1 Quality control requirements are included in the procedure, as follows:

18.1.1 DFTPP tune (section 14.3)

18.1.2 Initial Calibration (section 14.5)

18.1.3 Continuing Calibration (section 14.6)

18.1.4 In addition, quality control must include sections 18.3 through 18.7:

18.2 Blanks

18.2.1 After the DFTPP tune and calibration or continuing calibration have been run, a blank is required before sample analysis showing that the system is free from interferences and contamination at or below the reporting limit. The blank is a Method Preparation Blank (BLK) or a solvent instrument blank (IBL). An MPB is an extracted blank from a sample batch. An IBL is methylene chloride spiked only with internal standard. A BLK may be used for the blank if uncontaminated. Otherwise an IBL must be run. Run a BLK or IBL at least once each 12-hour shift. Run blanks more frequently if carryover is suspected from a high-concentration sample, or if laboratory contamination is in question. Extract a BLK for each batch of up to 20 samples. BLKs must be spiked with surrogates and carried through the same preparation as samples. Acceptance criteria for both the IBL and BLK are that all target analytes must have concentrations below the lowest reporting limit for samples analyzed that day. Common laboratory contaminants such as phthalates must have blank concentrations less than five times the reporting limit.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following the continuing calibration standard must be evaluated during or immediately after data acquisition. All internal standard retention times must be within  $\pm 30$  seconds of the current 12-hour continuing calibration standard. All internal standard quantitation ion areas must stay within a factor of two (-50% to +100%) of the mid-point standard from the initial calibration.

18.3.2 If at any time an internal standard fails the -50% to +100% area window, the ability to accurately quantitate analytes is reduced. Every effort must be made to prevent an internal standard failure, including dilution and reanalysis of samples. Refer to TriMatrix GR-04-101 for the corrective action sequence, should internal standards fail. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for a malfunction and appropriate repairs made. Once the problem is resolved, samples analyzed with the faulty spectrometer must be re-analyzed, with acceptable internal standard responses.

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#### 18.4 Surrogates

18.4.1 All samples must be spiked with surrogates. Until twenty samples of a given matrix have been analyzed, a default recovery window of 50% - 150% will be used. When twenty samples of a given matrix have been analyzed, in-house recovery limits will be generated (section 18.5.2-18.5.4) and used. Table 6 provides examples of in-house surrogate recovery limits. These are examples only. At a minimum, surrogate recovery limits must be updated annually, on a matrix-by-matrix basis.

18.4.2 Calculate upper and lower control limits for each surrogate, as follows:

Upper Control Limit (UCL) =  $p + 3s$

Lower Control Limit (LCL) =  $p - 3s$

where

$p$  = average percent recovery

$s$  = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If surrogate recovery is not within limits, take corrective action. Refer to TriMatrix GR-04-101 for the corrective action sequence, including re-extraction when sample volumes and holding times permit, and data qualification. If many surrogates are out-of-control for no apparent reason, the instrument must be inspected for a malfunction, and appropriate repairs made. When the problem is resolved, samples analyzed with the faulty instrument must be reanalyzed with acceptable surrogate recovery.

#### 18.5 Matrix Spikes and Laboratory Fortified Blanks

18.5.1 To assess extraction efficiency, a Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Blank Spike (BS) must be extracted. Spiking must contain the compounds in Table 7 and must be performed at least once every 20 samples for each matrix extracted. If only one to ten samples are analyzed in a month, at least one matrix spike, one matrix spike duplicate and one laboratory fortified blank is required. A BS must be extracted daily or with each sample batch, whichever is more frequent. Generally, the MPB and BS are run at the beginning of the 12-hour shift, and matrix spikes at the end. Running matrix spikes last helps document that the instrument is still operating correctly toward the end of the 12-hour shift.

NOTE: For State of Wisconsin samples and where project specifications require, all target analytes must be added for associated spikes, spike duplicates and BSs.

18.5.2 Until five matrix spikes, matrix spike duplicates, and laboratory fortified blanks have been analyzed, recoveries must be validated against default recovery limits of 50% - 150%. Matrix Spike/Matrix Spike Duplicate relative percent difference (RPD) limits must use a default maximum limit of 20%.

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18.5.3 When five data sets of a given matrix (water, soil, other) have been analyzed, calculate average percent recovery (R) and standard deviation of the average (SD). Express the recovery as the percent recovery interval from  $R \pm 3SD$ . For example, if  $R = 90\%$  and  $SD = 10\%$ , spike recovery limits are  $60\% - 120\%$ . Separate limits must be calculated for laboratory fortified blanks. The relative percent difference limit must be calculated by averaging five MS/MSD percent differences  $\pm 3$  standard deviations. Acceptance limits will be updated at least annually.

18.5.4 Calculate percent recovery as follows:

$$\% \text{ recovery} = \frac{(A_{\text{spk}} - A_{\text{smp}})}{\text{SPK}} \times 100$$

where:

$A_{\text{spk}}$  = The spiked sample concentration, in ug/L or mg/kg

$A_{\text{smp}}$  = The non-spiked sample concentration, in ug/L or mg/kg

$$\text{SPK} = \left( \frac{\text{concentration of spiked standard in ug/L} \times \text{mL spiked}}{\text{initial sample volume in L}} \right)$$

18.5.5 Calculate relative percent difference (RPD) as follows:

$$\% \text{ RPD} = \frac{\left| \frac{(MS - MSD)}{(MS + MSD)} \right|}{2} \times 100$$

where:

MS = The matrix spike concentration, in ug/L or mg/kg

MSD = The matrix spike duplicate concentration, in ug/L or mg/kg

18.5.6 If recovery or duplication are not within limits, refer to TriMatrix GR-04-101 for the corrective action sequence, to determine if or how data is qualified.

18.5.7 If a BS analyte is out-of-control, the problem must be immediately identified and corrected. Samples for the analyte must not be run until the problem is corrected. The purpose of a BS is to verify that out-of-control compounds in a MS or MSD are the result of matrix interference, rather than extraction or system error. Failure of any analyte in a BS, requires corrective action specific to the failed parameter. Every effort must be made to determine the cause for failure (mis-spiked, mis-extracted, or any other reason). When the problem is located, corrective action must be taken following TriMatrix SOP GR-04-101. If it is determined that re-extraction is necessary, the entire batch of samples must be re-extracted.

**NOTE:** If a BS recovery is biased high and there are no positive results for the analyte in the associated samples, results can be reported without the need for corrective action or qualification.

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## 18.6 Method Detection Limit Studies

- 18.6.1 A Method Detection Limit (MDL) study must be performed annually for every target compound analyzed. MDL studies must be performed on each instrument involved in sample analysis. Analyte results for which a study has not been performed must be reported as estimated. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. Minimum reporting limits are equal to the concentration spiked for the MDL study, provided the MDL passes. Reporting limits actually achieved for any given analysis will vary depending on instrument sensitivity, matrix, and extract dilutions. MDL studies must be run for aqueous and soil matrices.
- 18.6.2 The procedure followed for MDL studies is based on the method given in 40 Code of Federal Regulations, Part 136, Appendix B, latest revision. Method 8270C quality control governing the analysis must be followed.
- 18.6.3 Seven replicate extractions and subsequent analyses are performed using reagent water or Ottawa Sand spiked with the target compounds of interest at estimated minimum reporting limit concentrations.
- 18.6.4 For each target compound, standard deviation of the average is calculated and multiplied by 3.143. The resulting number is the calculated MDL.
- 18.6.5 If the concentration spiked is greater than or equal to the calculated MDL, and less than or equal to five times the calculated MDL, and there are no zero percent recoveries in the seven results, the MDL is acceptable. The MDL study must be repeated, only for the analyte(s) that did not pass. If a study needs repeated at a different concentration, the entire set of seven needs to be repeated. If a study does not pass due to poor reproducibility on one result, only that result needs repeated. Only one result may be rejected and repeated however. All seven results do not need to be obtained during the same 12-hour shift.
- 18.6.6 If a reporting limit is above a client or state-desired reporting limit, the calculated MDL may be used provided results are narrated. Narration must state that the reporting limit is the calculated MDL and when spiked at that level, analyte was not observed.
- 18.6.7 Table 6 is an MDL spreadsheet example. The spreadsheet is used to calculate and verify MDLs and reporting limits.

## 19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

- 19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time demonstration of capability study. While the demonstration of capability is not instrument dependent, a study is required on every instrument running samples, to demonstrate each instrument's ability to generate acceptable accuracy and precision.

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- 19.2 Prepare a 100 ug/L concentration of all compounds of interest in four 1000 mL aliquots of reagent water, by spiking with 0.100 mL of a 100 ng/uL standard. Extract to a 10 mL final volume. The spiking solution must be prepared independently from stock standards used for quantitation.
- 19.3 Analyze the four extracts using Method 8270C quality control, and following every step of the procedure. For each target compound, calculate average percent recovery "x" and standard deviation "s" in ug/L. Use all four results. Calculate by inputting into the IDC spreadsheet located on the laboratory intranet subdirectory "\\library\spreadsheets\training documents\qc\_idc-demonstration of capability(doc).xls".
- 19.3.1 For each target compound, "x" must pass the current ELEMENT BS acceptance limits. Also, relative standard deviation must be less than or equal to 20%. Calculate %RSD as  $[(s/x)*100]$ . If "s" and %RSD for all analytes pass both acceptance criteria, the demonstration of capability study is complete. The analyst and associated instrument may run samples. If %RSD exceeds 20% or "x" falls outside the current ELEMENT BS acceptance limits, analyst or instrument performance is unacceptable for the analyte.
- 19.3.2 When one or more analytes fail for "x" or %RSD, proceed according to section 19.4.
- 19.4 Locate and correct the source of any problem and repeat the study for failed analytes. Repeated failure however, will confirm a general problem with the procedure or technique used. If this occurs, locate and correct the problem, then repeat the study for all unacceptable compounds beginning with section 19.1. Samples may not be analyzed by any analyst or on any instrument, until a demonstration of capability study has been successfully completed. Copies of successful demonstration of capability studies, IDC spreadsheets and raw data must be given to Quality Assurance.

## **20.0 POLLUTION PREVENTION**

- 20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.
- 20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.
- 20.3 Conserve use of chemicals where applicable.
- 20.4 Comply with all environmental laws associated with chemicals in the laboratory.

## **21.0 WASTE MANAGEMENT**

- 21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.
- 21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.
- 21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

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## 22.0 REFERENCES

- 22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996*
- 22.2 *40 Code of Federal Regulations, most current edition, Pt. 136, App. A, Method 625-Base/Neutrals and Acids*
- 22.3 *40 Code of Federal Regulations, most current edition, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit*

## 23.0 ATTACHMENTS

- 23.1 Table 1 Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
- 23.2 Table 1A Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
- 23.3 Table 2 Default Reporting Limits
- 23.4 Table 2A Default Reporting Limits
- 23.5 Table 3 DFTPP Key Ions and Ion Abundance Criteria (EPA CLP)  
 Table 3A DFTPP Key Ions and Ion Abundance Criteria (Method 8270C Default Criteria)
- 23.6 Table 4 System Performance Check Compounds (SPCC)
- 23.7 Table 5 Continuing Check Compounds (CCC)
- 23.8 Table 6 Method Detection Limit Study Example (Partial Table)
- 23.9 Table 7 Demonstration of Capability Study Example (Partial Table)
- 23.10 Figure 1 Pentachlorophenol Tailing Factor
- 23.11 Figure 2 Continuing Calibration Standard Total Ion Chromatogram, Varian Ion Trap (10 ug/mL)
- 23.12 Figure 2A Continuing Calibration Standard Total Ion Chromatogram, Agilent 5973 MSD (10 ug/mL)
- 23.13 Figure 3 Standards Form Example
- 23.14 Figure 4 Analytical Run Logbook Example

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23.15 Method 8270C Selected Ion Monitoring (SIM) instructions

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**Attachment 23.1 (Table 1)**  
**Routine Target Compounds, Internal Standards, Surrogates, Retention Times,**  
**Quantitation and Secondary Ions**

Compound	Retention Time (min.) *	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d <sub>10</sub> (I.S.)	17.76	162	164, 160	3
Chrysene-d <sub>12</sub> (I.S.)	25.04	240	120, 236	5
Dichlorobenzene-1,4-d <sub>4</sub> (I.S.)	10.06	152	150, 115	1
Naphthalene-d <sub>8</sub> (I.S.)	13.21	136	68	2
Perylene-d <sub>12</sub> (I.S.)	29.83	264+265 (264)**	260, 265	6
Phenanthrene-d <sub>10</sub> (I.S.)	20.50	188	94,80	4
2-Fluorobiphenyl (surr.)	16.00	172	171	3
2-Fluorophenol (surr.)	7.18	112	64	1
Nitrobenzene-d <sub>5</sub> (surr.)	11.38	127+128 (128)**	82, 54	2
Phenol-d <sub>6</sub> (surr.)	9.13	99		1
2,4,6-Tribromophenol (surr.)	19.33	330	332, 141	3
Terphenyl-o (surr.)	21.03	230	215,229	5
Acenaphthene	17.84	152+154 (154)**	153, 152	3
Acenaphthylene	17.44	152	151, 153	3
Anthracene	20.63	178	176, 179	4
Benzoic acid	12.39	105	77,122	2
Benzo(a)anthracene	25.00	228	229, 226	5
Benzo(b and k)fluoranthene	28.24/28.40	252	253, 125	6
Benzo(g,h,i)perylene	37.32	276+277 (276)**	138, 277	6
Benzo(a)pyrene	29.60	252	253, 125	6
Benzyl alcohol	10.35	108	79, 77	1
Bis(2-chloroethoxy)methane	12.51	92+93 (93)**	95, 123	2
Bis(2-chloroethyl)ether	9.38	92+93 (93)**	63, 95	1
Bis(2-chloroisopropyl)ether	10.65	45	77, 121	1
Bis(2-ethylhexyl)phthalate	24.59	149	167, 279	5
4-Bromophenyl phenyl ether	19.73	248	250, 141	4
Butyl benzyl phthalate	23.56	149	91, 206	5
4-Chloroaniline	13.40	127	129	2
2-Chloronaphthalene	16.34	162	127, 164	3
4-Chloro-3-methylphenol	14.60	107	144, 142	2
2-Chlorophenol	9.57	128	64, 130	1
4-Chlorophenyl phenyl ether	18.87	204	206, 141	3

Approved By: <u>ms 1-22-09</u>	Approved By: <u>June 1/23/19</u>
QA Officer	Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
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**Attachment 23.1 (Table 1) - Continued**  
**Routine Target Compounds, Internal Standards, Surrogates, Retention Times,**  
**Quantitation and Secondary Ions**

Compound	Retention Time (min.) <sup>*</sup>	Quantitation Ion	Secondary Ion(s)	Internal Standard
Chrysene	25.10	228	226, 229	5
Dibenz(a,h)anthracene	35.57	278	139, 279	6
Dibenzofuran	18.20	168	139	3
Di-n-butylphthalate	21.23	149	150, 104	4
1,3-Dichlorobenzene	9.93	146	148, 111	1
1,4-Dichlorobenzene	10.10	146	148, 111	1
1,2-Dichlorobenzene	10.45	146	148, 111	1
3,3'-Dichlorobenzidine	24.80	252	254, 126	5
2,4-Dichlorophenol	12.84	162	164, 98	2
Diethylphthalate	18.59	149+177 (149)**	177, 150	3
2,4-Dimethylphenol	12.26	107	121, 122	2
Dimethylphthalate	17.03	163	194, 164	3
4,6-Dinitro-2-methylphenol	18.94	198	51, 105	4
2,4-Dinitrophenol	17.86	184	63, 154	3
2,4-Dinitrotoluene	18.12	165	63, 89	3
2,6-Dinitrotoluene	17.20	165	63, 89	3
1,2-Diphenylhydrazine	19.15	77	105, 182	4
Di-n-octylphthalate	26.32	149	167, 43	5
Fluoranthene	22.35	202	101, 203	4
Fluorene	18.89	166	165, 167	3
Hexachlorobenzene	19.87	284	142, 249	4
Hexachlorobutadiene	13.54	225	223, 227	2
Hexachlorocyclopentadiene	15.45	237	235, 272	3
Hexachloroethane	11.30	119	201, 199	1
Indeno(1,2,3-cd)pyrene	35.52	276+277 (276)**	138, 227	5
Isophorone	12.00	82	95, 138	2
2-Methylnaphthalene	15.06	142	141	2
2-Methylphenol (o-cresol)	10.58	108	79, 107	1
3- and/or 4-Methylphenol (m,p-cresol)	10.95	108	79, 107	1
Naphthalene	13.26	128	129, 127	2

Approved By: <u>MS 1-22-09</u>	Approved By: <u>mu 1/23/09</u>
QA Officer	Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
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**Attachment 23.1 (Table 1) - Continued**  
**Routine Target Compounds, Internal Standards, Surrogates, Retention Times,**  
**Quantitation and Secondary Ions**

Compound	Retention Time (min.) <sup>*</sup>	Quantitation Ion	Secondary Ion(s)	Internal Standard
2-Nitroaniline	16.59	138	92, 138	3
3-Nitroaniline	17.62	138	108, 92	3
4-Nitroaniline	18.90	108+138 (138)**	108, 92	3
Nitrobenzene	11.43	77+123 (77)**	65	2
2-Nitrophenol	12.21	109+139 (139) **	109, 65	2
4-Nitrophenol	18.02	65	109, 65	3
N-Nitrosodimethylamine	4.58	73+74 (74)**	42	1
N-Nitrosodiphenylamine	19.08	169	168, 167	4
N-Nitrosodipropylamine	10.95	70	42, 101, 1	1
Pentachlorophenol	20.18	266	264, 268	4
Phenanthrene	20.54	178	179, 176	4
Phenol	9.17	94	65, 66	1
Pyrene	22.74	202	200, 203	5
1,2,4-Trichlorobenzene	13.05	180	182, 145	2
2,4,5-Trichlorophenol	15.91	196	198, 200	3
2,4,6-Trichlorophenol	15.79	196	198, 200	3

I.S. = Internal Standard

surr. = Surrogate

\* retention time provided for example only.

\*\* for MSD

Approved By:                      1-22-09  
 QA Officer

Approved By:                      Jan 1/23/09  
 Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry Revision Number: 5.5  
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**Attachment 23.2 (Table 1A)**  
**Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions**

Compound	Retention Time (min.) <sup>*</sup>	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d <sub>10</sub> (I.S.)	17.05	162	160,164	3
Chrysene-d <sub>12</sub> (I.S.)	24.14	240	120,236	5
Dichlorobenzene-1,4-d <sub>4</sub> (I.S.)	9.39	152	150,115	1
Naphthalene-d <sub>8</sub> (I.S.)	12.49	136	68	2
Perylene-d <sub>12</sub> (I.S.)	28.22	264+265 (264)***	260	6
Phenanthrene-d <sub>10</sub> (I.S.)	19.96	188	94,80	4
Acetophenone	10.45	105	77,70	2
2-Acetylaminofluorene	23.74	181	223,180	3
4-Aminobiphenyl	18.63	169	168,167	3
Aniline	8.76	93	66,65	1
Aramite**	22.64/22.73/22.85	185	63,57	4
2-sec-Butyl-4,6-dinitrophenol	19.89	211	163,147	3
2,6-Dichlorophenol	12.69	162	164,63	2
p-(Dimethylamino)azobenzene	22.71	120	225,77	3
7,12-Dimethylbenz(a)anthracene	26.79	256	252,241	5
3,3'-Dimethylbenzidine	23.26	212	213,106	3
2,2-Dimethylphenethylamine	12.64	58	91	2
1,3-Dinitrobenzene	16.55	76	139,169,50, 168	2
Carbazole	17.97	167	83, 139	4
Ethyl methacrylate	4.98	69	41,99	1
Ethyl methanesulfonate	7.74	79	109,97	1
Hexachlorophene	27.48	196	198,209	6
Hexachloropropene	12.75	213	215,211	2
Isosafrole	15.58	162	131,104	2
Methapyrilene	21.39	58	97,72	3
3-Methylcholanthrene	29.28	268	126,252	5
Methyl methacrylate	3.67	69	41,100	1
Methyl methanesulfonate	6.34	80	65,79	1
1,4-Naphthoquinone	16.23	158	104,102	2
1-Naphthylamine	17.87	143	115,116	2
2-Naphthylamine	18.05	143	115,116	2
4-Nitroquinoline-1-oxide	21.38	190	89,101	4

Approved By: QA 1-22-09 QA Officer Approved By: Jan 1/23/18 Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
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**Attachment 23.2 (Table 1A) - Continued**  
**Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions**

Compound	Retention Time (min.) <sup>*</sup>	Quantitation Ion	Secondary Ion(s)	Internal Standard
N-Nitrosodi-n-butylamine	13.64	84	57,41	2
N-Nitrosodiethylamine	7.14	102	42,44	1
N-Nitrosomethylethylamine	5.75	88	42,43	1
N-Nitrosomorpholine	10.48	56	116,86	2
N-Nitrosopiperidine	11.24	114	42,56	2
N-Nitrosopyrrolidine	10.39	100	41,42	1
5-Nitro-o-toluidine	18.42	152	106,77	3
Pentachlorobenzene	17.46	250	252,248	3
Pentachloroethane	8.74	167	93,117	2
Pentachloronitrobenzene	19.78	237	169,142	4
Phenacetin	19.17	108	109,179	3
p-Phenylenediamine	13.80	108	80,107	2
2-Picoline	6.73	93	66,92	1
Pronamide	20.20	173	175,145	3
Pyridine	6.32	79	52,51	1
Safrole	14.24	162	131,101	2
1,2,4,5-Tetrachlorobenzene	14.76	216	214,218	3
2,3,4,6-Tetrachlorophenol	17.83	232	230,234	3
o-Toluidine	10.54	107	106,79	2
sym-Trinitrobenzene	19.09	75	74,213	3
Octachlorocyclopentene (C-58)	19.03	307+309	237	3

I.S. = Internal Standard

\* retention time provided for example only

\*\* Aramite chromatographs into three peaks

\*\*\* For MSD

Approved By: MD 1-22-09  
 QA Officer

Approved By: JMC 1/23/09  
 Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
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**Attachment 23.3 (Table 2)**  
**Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Acenaphthene	83-32-9	5	0.33
Acenaphthylene	208-96-8	5	0.33
Anthracene	120-12-7	5	0.33
Benzoic acid	65-85-0	50	3.3
Benzo(a)anthracene	56-55-3	5	0.33
Benzo(b and k)fluoranthene	205-99-2 / 207-08-9	5	0.33
Benzo(g,h,i)perylene	191-24-2	5	0.33
Benzo(a)pyrene	50-32-8	5	0.33
Benzyl alcohol	100-51-6	50	1.3
Bis(2-chloroethoxy)methane	111-91-1	5	0.33
Bis(2-chloroethyl)ether	111-44-4	5	0.33
Bis(2-chloroisopropyl)ether	39638-32-9	5	0.33
Bis(2-ethylhexyl)phthalate	117-81-7	5	0.33
4-Bromophenyl phenyl ether	101-55-3	5	0.33
Butyl benzyl phthalate	85-68-7	5	0.33
Carbazole	86-74-8	5	0.33
4-Chloroaniline	106-47-8	20	1.3
2-Chloronaphthalene	91-58-7	5	0.33
4-Chloro-3-methylphenol	59-50-7	5	0.33
2-Chlorophenol	95-57-8	5	0.33
4-Chlorophenyl phenyl ether	7005-72-3	5	0.33
Chrysene	218-01-9	5	0.33
Dibenz(a,h)anthracene	53-70-3	5	0.33
Dibenzofuran	132-64-9	5	0.33
Di-n-butylphthalate	84-74-2	5	0.33
1,3-Dichlorobenzene	541-73-1	5	0.33
1,4-Dichlorobenzene	106-46-7	5	0.33
1,2-Dichlorobenzene	95-50-1	5	0.33
3,3'-Dichlorobenzidine	91-94-1	20	2.0
2,4-Dichlorophenol	120-83-2	5	0.33
Diethylphthalate	84-66-2	5	0.33

Approved By: <u>          m          1-22-09          </u>	Approved By: <u>          JMK 1/23/18          </u>
QA Officer	Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
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**Attachment 23.3 (Table 2) - Continued**  
**Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
2,4-Dimethylphenol	105-67-9	5	0.33
Dimethylphthalate	131-11-3	5	0.33
4,6-Dinitro-2-methylphenol	534-52-1	20	1.7
2,4-Dinitrophenol	51-28-5	20	1.7
2,4-Dinitrotoluene	121-14-2	5	0.33
2,6-Dinitrotoluene	606-20-2	5	0.33
Diphenylamine	122-39-4	5	0.33
Di-n-octylphthalate	117-84-0	5	0.33
Fluoranthene	206-44-0	5	0.33
Fluorene	86-73-7	5	0.33
Hexachlorobenzene	118-74-1	5	0.33
Hexachlorobutadiene	87-68-3	5	0.33
Hexachlorocyclopentadiene	77-47-4	5	0.33
Hexachloroethane	67-72-1	5	0.33
Indeno(1,2,3-cd)pyrene	193-39-5	5	0.33
Isophorone	78-59-1	5	0.33
2-Methylnaphthalene	91-57-6	5	0.33
2-Methylphenol (o-cresol)	95-48-7	5	0.33
3- and/or 4-Methylphenol (m,p-cresol)	106-44-5	5	0.33
Naphthalene	91-20-3	5	0.33
2-Nitroaniline	88-74-4	20	1.7
3-Nitroaniline	99-09-2	20	1.7
4-Nitroaniline	100-01-6	20	1.7
Nitrobenzene	98-95-3	5	0.33
2-Nitrophenol	88-75-5	5	0.33
4-Nitrophenol	100-02-7	20	1.7
N-Nitrosodimethylamine	62-75-9	5	0.33
N-Nitrosodiphenylamine	86-30-6	5	0.33
N-Nitrosodipropylamine	621-64-7	5	0.33
Pentachlorophenol	87-86-5	20	1.7

Approved By: RD 1-22-09  
 QA Officer

Approved By: John 1/23/9  
 Area Supervisor



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**Attachment 23.3 (Table 2) - Continued**  
**Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Phenanthrene	85-01-8	5	0.33
Phenol	108-95-2	5	0.33
Pyrene	129-00-0	5	0.33
1,2,4-Trichlorobenzene	120-82-1	5	0.33
2,4,5-Trichlorophenol	95-95-4	5	0.33
2,4,6-Trichlorophenol	88-06-2	5	0.33

\* Sample reporting limits are highly matrix-dependent. The reporting limits listed are provided for guidance and may not always be achievable.

\*\* Water reporting limits are based on an extraction of 1000 mL to 10 mL, in the MDL study. Reporting limits listed for soil/sediment are based on an extraction of 30 g to 10 mL.

Approved By: <u>          <i>MD</i> 1-22-09          </u> QA Officer	Approved By: <u>          <i>JVC</i> 1/23/19          </u> Area Supervisor
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**Attachment 23.4 (Table 2A)**  
**Additional Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Acetophenone	98-86-2	10	0.33
2-Acetylaminofluorene	53-96-3	50	2.0
4-Aminobiphenyl	92-67-1	10	0.33
Aniline	62-53-3	5	1.7
Aramite	140-57-8	50	2.0
2-sec-Butyl-4,6-dinitrophenol	88-85-7	5	2.0
2,6-Dichlorophenol	87-65-0	5	0.33
p-(Dimethylamino)azobenzene	60-11-7	10	0.33
7,12-Dimethylbenz(a)anthracene	57-97-6	10	0.33
3,3'-Dimethylbenzidine	119-93-7	50	2.0
2,2-Dimethylphenethylamine	122-09-8	20	0.70
1,3-Dinitrobenzene	99-65-0	5	0.33
Ethyl methacrylate	97-63-2	50	2.0
Ethyl methanesulfonate	62-50-0	10	1.0
Hexachlorophene	70-30-4	***	***
Hexachloropropene	1888-71-7	50	2.0
Isosafrole	120-58-1	20	0.70
Methapyrilene	91-80-5	10	1.0
3-Methylcholanthrene	56-49-5	50	2.0
Methyl methacrylate	80-62-6	50	2.0
Methyl methanesulfonate	66-27-3	50	2.0
1,4-Naphthoquinone	130-15-4	1000	30
1-Naphthylamine	134-32-7	50	2.0
2-Naphthylamine	91-59-8	50	2.0
4-Nitroquinoline-1-oxide	56-57-5	500	20
N-Nitrosodi-n-butylamine	924-16-3	20	0.90
N-Nitrosodiethylamine	55-18-5	5	2.0
N-Nitrosomethylethylamine	62-75-9	5	2.0
N-Nitrosomorpholine	59-89-2	20	0.70
N-Nitrosopiperidine	100-75-4	20	0.70

Approved By: <u>          JD 1-22-09          </u> QA Officer	Approved By: <u>          Jme 1/23/09          </u> Area Supervisor
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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry EPA Method 625, SW-846 Method 8270C SOP Number: <b>GR-04-103</b>	Revision Number: 5.5 Date Revised: 1/22/09 Date Initiated: 12/9/98
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**Attachment 23.4 (Table 2A) - Continued**  
**Additional Default Reporting Limits**

Semivolatiles	CAS Number	Default Reporting Limits**	
		Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
N-Nitrosopyrrolidine	930-55-2	20	0.70
5-Nitro-o-toluidine	99-55-8	10	0.33
Pentachlorobenzene	608-93-5	5	0.33
Pentachloroethane	76-01-7	50	2.0
Pentachloronitrobenzene	82-68-8	20	0.50
Phenacetin	62-44-2	10	0.33
p-Phenylenediamine	106-50-3	***	***
2-Picoline	109-06-8	20	0.70
Pronamide	23950-58-5	10	0.33
Pyridine	110-86-1	10	0.33
Safrole	94-59-7	10	0.33
1,2,4,5-Tetrachlorobenzene	95-94-3	10	0.50
2,3,4,6-Tetrachlorophenol	58-90-2	50	2.0
o-Toluidine	95-53-4	10	0.33
sym-Trinitrobenzene	99-35-4	20	0.70
Octachlorocyclopentene (C-58)	706-78-5	0.1	0.02

- \* Sample reporting limits are highly matrix-dependent. The listed values are provided for guidance and may not always be achievable.
- \*\* Water reporting limits are based on extracting 1000 mL to 1.0 mL, in the MDL study. Reporting limits listed for soil/sediment are based on extracting 30 g to 1.0 mL.
- \*\*\* These compounds are demonstrated to be difficult to extract from water, or difficult to chromatograph. Method detection limits are not available.

Approved By: <u>          <i>PA</i> 1-22-09          </u> QA Officer	Approved By: <u>          <i>John</i> 1/23/09          </u> Area Supervisor
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**Attachment 23.5 (Table 3)**  
**DFTPP Key Ions and Ion Abundance Criteria**  
**(EPA CLP)**

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	25-75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance (see note)
199	5-9% of mass 198
275	10-30% of mass 198
365	>0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198 which is the nominal base peak, even though ion abundances of m/z 442 may be up to 110% that of m/z 198.

**Attachment 23.5 (Table 3A)**  
**DFTPP Key Ions and Ion Abundance Criteria**  
**(Method 8270C Defaults)**

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Approved By:  1-22-09 QA Officer	Approved By:  1/23/19 Area Supervisor
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**Attachment 23.6 (Table 4)**  
**System Performance Check Compounds (SPCC)**

Base/Neutral Fraction	Acid Fraction
N-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

**Attachment 23.7 (Table 5)**  
**Calibration Check Compounds (CCC)**

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Approved By: <u>          <i>ms</i> 1-22-09          </u> QA Officer	Approved By: <u>          <i>pm</i> 1/23/09          </u> Area Supervisor
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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
EPA Method 625, SW-846 Method 8270C  
SOP Number: GR-04-103

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Revision Number: 5.5  
Date Revised: 1/22/09  
Date Initiated: 12/9/98

Attachment 23.8 (Table 6)  
Method Detection Limit Study Example



SEMI-VOLATILE LABORATORY  
INSTRUMENT NUMBER 195 2004 WATER  
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
N-Nitrosodimethylamine	8270C	12/29/2003	1.00	ug/L	0.550	0.490	0.370	0.180	0.710	0.620	0.310	0.461	46%	0.185	0.582
Phenol	8270C	12/31/2003	0.500	ug/L	0.273	0.264	0.270	0.340	0.325	0.343	0.281	0.299	60%	0.0350	0.110
Aniline	8270C	12/29/2003	1.00	ug/L	0.450	0.400	0.330	0.260	0.550	0.470	0.290	0.393	39%	0.105	0.330
Bis (2-chloroethyl) ether	8270C	12/31/2003	0.500	ug/L	0.397	0.466	0.483	0.537	0.501	0.429	0.514	0.475	95%	0.0490	0.154
2-Chlorophenol	8270C	12/31/2003	0.500	ug/L	0.425	0.372	0.411	0.476	0.460	0.482	0.431	0.437	87%	0.0391	0.123
1,3-Dichlorobenzene	8270C	12/31/2003	0.500	ug/L	0.411	0.377	0.393	0.465	0.456	0.457	0.424	0.426	85%	0.0344	0.108
1,4-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.01	0.910	0.780	0.580	1.06	0.880	0.620	0.834	83%	0.184	0.578
Benzaldehyde	8270C	12/29/2003	1.00	ug/L	0.880	0.790	0.900	0.710	0.980	1.02	0.770	0.864	86%	0.114	0.357
Benzyl Alcohol	8270C	12/29/2003	1.00	ug/L	0.490	0.420	0.320	0.280	0.690	0.540	0.330	0.439	44%	0.146	0.458
1,2-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.04	0.870	0.740	0.590	1.10	0.870	0.610	0.831	83%	0.198	0.621
2-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.488	0.454	0.521	0.416	0.516	0.539	0.400	0.476	95%	0.0542	0.170
Bis-(2-chloroisopropyl)-ether	8270C	12/31/2003	0.500	ug/L	0.512	0.476	0.496	0.701	0.578	0.519	0.502	0.541	108%	0.0775	0.244
Acetophenone	8270C	12/29/2003	1.00	ug/L	0.810	0.720	0.710	0.700	0.860	0.840	0.670	0.759	76%	0.0760	0.239
4-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.345	0.422	0.474	0.451	0.449	0.435	0.409	0.426	85%	0.0416	0.131
N-Nitrosodi-n-propylamine	8270C	12/29/2003	1.00	ug/L	1.16	0.780	0.640	0.570	1.12	0.860	0.600	0.819	82%	0.242	0.761
Hexachloroethane	8270C	12/29/2003	1.00	ug/L	0.920	0.830	0.650	0.480	0.940	0.760	0.550	0.733	73%	0.179	0.563
Nitrobenzene	8270C	12/31/2003	0.500	ug/L	0.450	0.528	0.449	0.609	0.542	0.491	0.466	0.505	101%	0.0586	0.184
Isophorone	8270C	12/31/2003	0.500	ug/L	0.532	0.481	0.563	0.619	0.641	0.547	0.601	0.569	114%	0.0553	0.174
2-Nitrophenol	8270C	12/31/2003	0.500	ug/L	0.526	0.604	0.636	0.554	0.654	0.596	0.647	0.602	120%	0.0483	0.152
2,4-Dimethylphenol	8270C	12/31/2003	0.500	ug/L	0.367	0.315	0.375	0.295	0.374	0.398	0.364	0.355	71%	0.0366	0.115
Benzoic Acid	8270C	2/11/2004	5.00	ug/L	3.36	3.78	3.23	1.52	3.48	3.39	3.43	3.17	63%	0.747	2.35
Bis(2-chloroethoxy)methane	8270C	12/31/2003	0.500	ug/L	0.469	0.399	0.513	0.550	0.477	0.510	0.484	0.486	97%	0.0472	0.148
2,4-Dichlorophenol	8270C	12/29/2003	1.00	ug/L	0.840	0.710	0.530	0.440	0.900	0.840	0.510	0.681	68%	0.187	0.587
1,2,4-Trichlorobenzene	8270C	12/29/2003	1.00	ug/L	0.970	0.820	0.650	0.500	1.00	0.800	0.560	0.757	76%	0.194	0.611

Approved By: PD 1-22-09  
QA Officer

Approved By: jmw 1/23/09  
Area Supervisor





SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
EPA Method 625, SW-846 Method 8270C  
SOP Number: GR-04-103

Revision Number: 5.5  
Date Revised: 1/22/09  
Date Initiated: 12/9/98

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Attachment 23.9 (Table 7)  
Demonstration of Capability Study Example



SEMI-VOLATILE LABORATORY  
DEMONSTRATION OF CAPABILITY FOR SCOTT J. PUGH

Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/Fail
Phenol	12/15/2003	8270c	133	ug/L	10.0000	3.9900	3.9300	4.3400	4.0800	40.9%	19 -55	PASS	4.4%	20	PASS	PASS
Aniline	12/15/2003	8270c	133	ug/L	10.0000	5.2300	5.3400	5.8100	5.5100	54.7%	23 -84	PASS	4.6%	20	PASS	PASS
Bis(2-Chloroethyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	8.9700	8.8900	9.6100	9.3900	92.2%	70 -130	PASS	3.7%	20	PASS	PASS
2-Chlorophenol	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9400	8.8800	8.2500	82.2%	70 -130	PASS	5.8%	20	PASS	PASS
1,3-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.4600	7.8600	8.2400	7.8000	78.4%	70 -130	PASS	4.1%	20	PASS	PASS
1,4-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.1900	7.6200	8.2100	7.7500	76.9%	70 -130	PASS	5.5%	20	PASS	PASS
Benzyl Alcohol	12/15/2003	8270c	133	ug/L	10.0000	8.1300	8.2300	8.6500	8.7800	84.5%	70 -130	PASS	3.7%	20	PASS	PASS
1,2-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.8900	8.0300	8.6100	8.2500	82.0%	70 -130	PASS	3.8%	20	PASS	PASS
2-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.5500	7.6700	8.0200	7.7800	77.6%	70 -130	PASS	2.6%	20	PASS	PASS
Bis(2-Chloroisopropyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	9.3000	9.3700	9.7200	9.5600	94.9%	70 -130	PASS	2.0%	20	PASS	PASS
4-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2900	7.1100	7.9500	7.7100	75.2%	70 -130	PASS	5.1%	20	PASS	PASS
N-Nitrosodi-N-Propylamine	12/15/2003	8270c	133	ug/L	10.0000	9.3500	9.2300	10.4000	10.0200	97.5%	70 -130	PASS	5.7%	20	PASS	PASS
Hexachloroethane	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9000	8.3300	7.8200	79.6%	70 -130	PASS	3.1%	20	PASS	PASS
Nitrobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.8400	9.3700	9.4300	8.9500	91.5%	70 -130	PASS	3.2%	20	PASS	PASS
Isophorone	12/15/2003	8270c	133	ug/L	10.0000	9.3100	9.7400	9.3600	9.5900	95.0%	70 -130	PASS	2.1%	20	PASS	PASS
2-Nitrophenol	12/15/2003	8270c	133	ug/L	10.0000	8.8100	9.5200	9.5200	9.3600	93.0%	70 -130	PASS	3.6%	20	PASS	PASS
2,4-Dimethylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2100	7.2400	7.0800	6.8600	71.0%	70 -130	PASS	2.4%	20	PASS	PASS
Benzoic Acid	12/15/2003	8270c	133	ug/L	10.0000	4.3400	5.4500	5.3600	5.5800	51.8%	20 -69	PASS	11.0%	20	PASS	PASS
Bis(2-Chloroethoxy)Methane	12/15/2003	8270c	133	ug/L	10.0000	9.1600	9.5800	9.5900	9.2700	94.0%	70 -130	PASS	2.3%	20	PASS	PASS
2,4-Dichlorophenol	12/15/2003	8270c	133	ug/L	10.0000	9.0800	9.3200	9.4000	9.3400	92.9%	70 -130	PASS	1.5%	20	PASS	PASS
1,2,4-Trichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.2000	8.6700	9.0400	8.5100	86.1%	70 -130	PASS	4.1%	20	PASS	PASS
Naphthalene	12/15/2003	8270c	133	ug/L	10.0000	9.2200	9.6100	9.3200	8.9200	92.7%	70 -130	PASS	3.1%	20	PASS	PASS

Approved By: 10 1-22-09  
QA Officer

Approved By: 1/22/09  
Area Supervisor





Revision Number: 5.5  
Date Revised: 1/22/09  
Date Initiated: 12/9/98



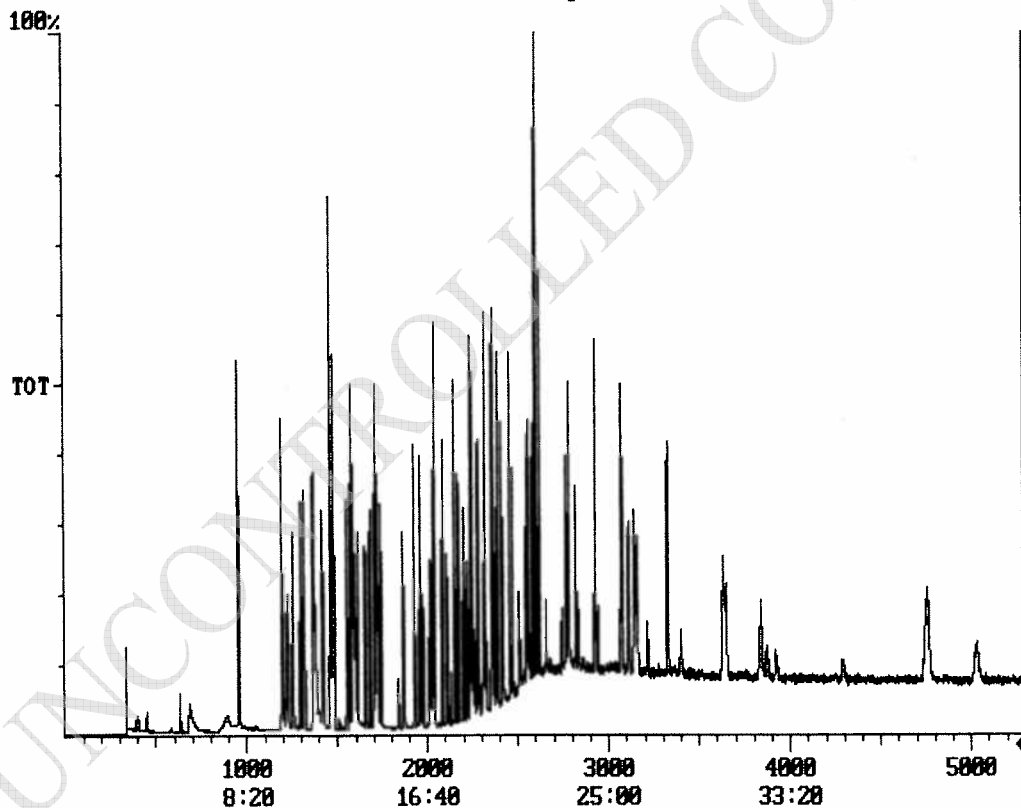
SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
EPA Method 625, SW-846 Method 8270C  
SOP Number: **GR-04-103**


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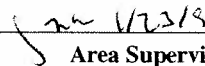
Revision Number: 5.5  
Date Revised: 1/22/09  
Date Initiated: 12/9/98

**Attachment 23.11 (Figure 2)**  
**Continuing Calibration Standard (10 ug/mL)**  
**Total Ion Chromatogram, Varian Ion Trap**

Chromatogram Plot C:\SATURN\DATA\18C8428A Date: 04/28/04 12:04:14  
Comment: INSTRUMENT 133  
Scan: 5288 Seg: 1 Group: 0 Retention: 44:00 RIC: 977062 Masses: 43-433  
Plotted: 1 to 5288 Range: 1 to 5288 100% = 12608842



Approved By:  1-22-09  
QA Officer

Approved By:  1/23/9  
Area Supervisor





SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry  
EPA Method 625, SW-846 Method 8270C  
SOP Number: **GR-04-103** page 43 of 46

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Date Initiated: 12/9/98

**Attachment 23.12 (Figure 2A)**  
**Continuing Calibration Standard (10 ug/mL)**  
**Total Ion Chromatogram, Agilent 5973 MSD**

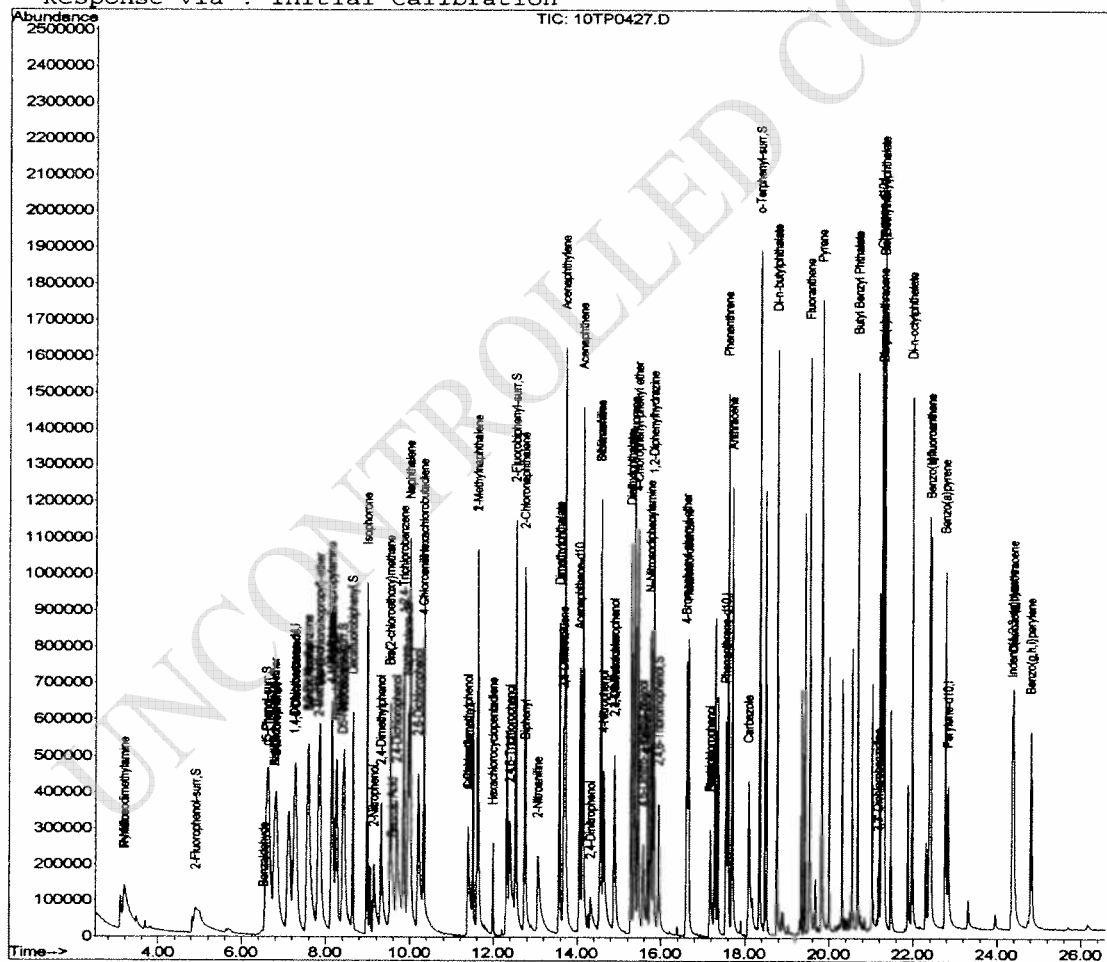
## Quantitation Report

Data File : C:\HPCHEM\1\DATA\04-27-04\10TP0427.D  
Acq On : 27 Apr 2004 10:17  
Sample : 10ppm bna pest  
Misc :  
MS Integration Params: rteint.p  
Quant Time: Apr 27 10:44 2004 Quant

Vial: 74  
Operator:  
Inst : GC/MS Ins  
Multiplr: 1.00

Quant Results File: BNA04074.RES

Method : C:\HPCHEM\1\METHODS\BNA04074.M (RTE Integrator)  
Title : 8270C/625 BNA  
Last Update : Tue Apr 27 17:24:56 2004  
Response via : Initial Calibration



10TP0427.D BNA04074.M

Thu Apr 29 14:34:16 2004

Page 4

Approved By: POS 1-22-09  
QA Officer

Approved By: mu 1/23/9  
Area Supervisor



SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry      Revision Number: 5.5  
 EPA Method 625, SW-846 Method 8270C      Date Revised: 1/22/09  
 SOP Number: **GR-04-103**      page 44 of 46      Date Initiated: 12/9/98

**Attachment 23.13 (Figure 3)**  
**Standards Form Example**

**Analytical Standard Record**

**TriMatrix Laboratories, Inc.**

**A605395**


Description:	DFTPP Tune Standard	Expires:	Aug-27-06
Standard Type:	MS Tune	Prepared:	May-11-06
Solvent:	Solvent Lot # C07H05	Prepared By:	Scott J. Pugh
Final Volume (mls):	1	Department:	Semivolatiles MS
Vials:	1	Last Edit:	May-11-06 09:04 by SJP

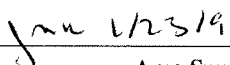
Solvent: MeCl

Analyte	CAS Number	Concentration	Units
4,4'-DDT	50-29-3	50	ppm
Benzidine	92-87-5	50	ppm
Decafluorotriphenylphosphine		5	ppm
Pentachlorophenol	87-86-5	50	ppm

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A409098	Pentachlorophenol, SVMS	Aug-27-01	** Vendor **	Aug-27-06	Dec-01-05 11:57 by RGJ	0.05
A512731	4,4-DDT SVMS	Dec-08-05	** Vendor **	Dec-08-06	Dec-27-05 09:53 by SJP	0.05
A601262	Benzidine, SVMS	Feb-24-05	** Vendor **	Feb-24-08	Jan-09-06 15:23 by SJP	0.05
A603057	DFTPP, SVMS	Feb-27-06	** Vendor **	Feb-27-07	Mar-02-06 08:58 by SJP	0.005

Approved By:  1-22-09  
 QA Officer

Approved By:  1/23/09  
 Area Supervisor





Revision Number: 5.5  
Date Revised: 1/22/09  
Date Initiated: 12/9/98

**Attachment 23.14 (Figure 4)**  
**Analytical Run Logbook Example**



Instrument ID: Saturn GC/MS Instrument #133		Date: 4/23/04	Taped: _____		Disk ID: _____	Analyst: SP5		
Instrument Settings		Acquisition Information		Temperature Program				Miscellaneous
Multiplier: 1550	Scan Range: 40-450 amu	GC	Temp. (C)	Rate (C/min)	Time (min)	Total (min)	New Column Date: 4/6/4	
AGC 7000	Seconds/Scan: 0.500						New Septa: Yes (No)	
Interface Temp: 250C	Filament Delay: 3.5 min.		Step #1	45	0.0	1.5	1.5	Working Std. #: 54.33-9
Source Temp: 190C	Threshold: 2 counts		Step #2	200	10	15.5	17.0	Int Std. #: 54.31.5
Head Pressure: 8 psi	Mass Defect: 0	Step #3	315	25	4.6	21.6	Tune Std. #: 54.23-8	
	Background Mass: 35 amu	Step #4	315	0.0	16.4	38	Inj. Vol.: 1 uL	
	Curve Date: 1/30/14	Inject.	45	0.0	0.5	0.5	DFTPP Area 198: 993078	
		Inject.	300	100	2.55	3.5	DDT Breakdown: 0%	
		Inject.	300	0.0	15	15.05		
Sample / File ID	Client	Inj Time	Dil	Matrix	Fraction	Method	Notes	
DFT0423C		8:47		QC	DFTPP	8270	Passed	
5C0423A		9:21		↓	BNA		Passed	
BLK0423A		10:10	STR	QC				
358328		11:22		WTR				
358329		12:14						
358330		13:05						
358331		13:45						
358332		14:25						
358333		15:01						
358334		15:49						
358335		16:24						
358336		17:01	STR	WTR	BNA	8270		

revision: 1.0

Approved By: Jue 1/23/19  
Area Supervisor



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SOP Name: Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry	Revision Number: 5.5
EPA Method 625, SW-846 Method 8270C	Date Revised: 1/22/09
SOP Number: <b>GR-04-103</b>	Date Initiated: 12/9/98

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**Attachment 23.15**  
**Method 8270C Selected Ion Monitoring (SIM) Instructions**

Selected Ion Monitoring (SIM) allows the mass spectrometer to detect specific compounds with very high sensitivity. In SIM mode the instrument obtains data at select masses of interest instead of stepping the mass filter over a wide mass range.

To set up SIM monitoring, the compounds of interest must first be analyzed by acquiring a full GCMS scan to determine the ions to use. Two to three ions are selected for each target compound. Include the most abundant ion if it is unique to the compound. Higher mass ions are usually more specific and separated from interference.

After choosing spectral fragments (ions) to monitor, an accurate mass must be determined ( $\pm 0.1$  AMU). The easiest way to determine mass accuracy is by tabulating the full-scan spectra of the compound. Print the mass table and use the  $m/z$  value in the tabulation to the nearest 0.1 AMU for the SIM acquisition mass.

After SIM acquisition masses are determined for each compound, ions are grouped according to elution time. Limit the number of ions being monitored in a group to between 3 and 8. Only one group can be monitored at a time. The end-time for one group becomes the start-time for the next group. Using these groups, the important ions for a list of compounds can be monitored by switching from one group to another at the appropriate time during a chromatographic run.

Once the monitoring ions are determined and the ions grouped, set a dwell time for each group to optimize the cycle time. Obtain 15 to 20 scans across each peak. A dwell time of 50 msec for each group is a good starting point. Acquire a run with this dwell time and note the number of scans across each peak. If the number of scans is less than 10, the dwell time needs reduced. If more than 25 scans, the dwell time needs increased.

Follow every step in the analytical procedure when using the SIM. A full-scan tune is acquired and must pass at the start of each 12-hour shift. Use a calibration curve of at least five points that pass acceptance criteria. Curve concentrations will depend on compound response and client requirements. Depending upon response, the low calibration standard is generally between 0.002 ug/mL and 0.1 ug/mL. Internal standard concentration is 0.5 ug/mL.

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Approved By:     *JD* 1-22-09      
QA Officer

Approved By:     *Jm* 1/23/09      
Area Supervisor





## STANDARD OPERATING PROCEDURE

### Extraction of Base Neutrals and Acids from Water

EPA Method 625  
SW-846 Method 3510C

#### APPROVALS:

Area Supervisor: Brian J. Hall Date: 2/12/09  
Brian J. Hall  
QA Officer: Tom C. Booher Date: 2-12-09  
Tom C. Booher  
Operations Manager: Jeff P. Glaser Date: 2/12/09  
Jeff P. Glaser

Procedure Number: GR-09-101  
Revision Number: 3.3

Date Initiated: 1/18/96  
Effective Date: 3/12/09

Date Revised: 2/12/09  
Pages Revised: All

By: Dan J. Mierendorf  
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
<u>4/16/10</u>	<u>Brian Hall</u>	<u>4/16/11</u>
_____	_____	_____
_____	_____	_____



SOP Name: Extraction of Base Neutrals and Acids from Water  
EPA Method 625, SW-846 Method 3510C  
SOP Number: GR-09-101

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Revision Number: 3.3  
Date Revised: 2/12/09  
Date Initiated: 1/18/96

## 1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes extraction of insoluble and slightly soluble semi-volatile organic base-neutral and acid compounds from aqueous samples and TCLP leachates. Also described are concentration techniques preparatory to GC/MS analysis.
- 1.2 The procedure is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by performance of a satisfactory demonstration of capability study.

## 2.0 PRINCIPLE METHOD REFERENCES

- 2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition*. Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"
- 2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

## 3.0 SUMMARY OF PROCEDURE

- 3.1 A measured sample volume, usually 1 L, is serially extracted with methylene chloride at acidic pH, then again at basic pH using separatory funnels.
- 3.2 Extracts are dried and concentrated to a final volume of 1.0 mL or 10 mL before GCMS analysis.

## 4.0 PARAMETER OR COMPOUND LIST

- 4.1 Refer to Table 1 in TriMatrix SOP GR-04-103 for a compound list associated with this procedure.

## 5.0 REFERENCED SOPs

- 5.1 TriMatrix SOP GR-04-103, *Semi-Volatile Laboratory Ion Trap Mass Spectrometry Analysis of Base/Neutral/Acid Compounds*, latest revision
- 5.2 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision
- 5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision
- 5.4 TriMatrix SOP GR-16-100, *Organic Glassware Cleaning*, latest revision
- 5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

## 6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

Approved By: ms 2-12-09  
QA Officer

Approved By: Bjt 2/12/09  
Area Supervisor



SOP Name: Extraction of Base Neutrals and Acids from Water  
EPA Method 625, SW-846 Method 3510C  
SOP Number: GR-09-101

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Revision Number: 3.3  
Date Revised: 2/12/09  
Date Initiated: 1/18/96

- 6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware. Contamination leads to discrete artifacts and elevated baselines. A method performance blank (MPB) must be performed routinely, to demonstrate that equipment and reagents are not contaminated.
- 6.2 Always use high purity reagents, solvents and clean glassware, cleaned in accordance with TriMatrix SOP GR-16-100.
- 6.3 Matrix interference is co-extracted with analyte from samples. Extraction interferences will vary considerably from matrix to matrix.
- 6.4 Phthalate esters co-elute during analysis as extraneous chromatographic peaks. Do not use any type of flexible plastic during extraction. Contact with extraction solvents will leach phthalate esters into sample extracts, presenting interference in compound identification and quantitation.
- 6.5 Matrix interference can also affect analyte recovery. Re-extraction may be necessary if surrogates or other spiked compounds fail established laboratory recovery limits.

## 7.0 SAFETY PRECAUTIONS

- 7.1 Extraction personnel must wear lab coats and approved safety glasses while in the prep lab area. In addition, disposable elastic gloves must be worn whenever samples, solvents or reagents are handled.
- 7.2 For extractions and surrogate/spike/reagent solution preparations, follow all safety instructions outlined in the latest revision of the Chemical Hygiene Plan and Laboratory Safety Manual.
- 7.3 For proper spill response, chemical storage and waste disposal refer to the latest revision of TriMatrix SOP GR-15-102.
- 7.4 Toxicity and carcinogenicity of materials used in this procedure have not been completely and precisely defined.
- 7.4.1 All chemicals must be treated as a potential health hazard.
- 7.4.2 Exposure can be reduced to the lowest possible level by adherence to established safety practices.
- 7.4.3 Material Safety Data Sheets are maintained within the laboratory (in the intranet library) of all chemicals used in this procedure.
- 7.5 Safety issues must be brought to the immediate attention of the area supervisor.

## 8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

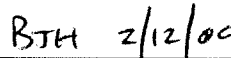
- 8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

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QA Officer

Approved By: \_\_\_\_\_



Area Supervisor



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- 8.2 All aqueous samples must be extracted within 7 days of the collection date. TCLP leachates must be extracted within 7 days of complete leachate generation. Extract analysis must occur within 40 days of the extraction.
- 8.3 When not in use, samples must be stored in the walk-in cooler at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , or as specified by the project.
- 8.4 Analysts must use care when handling sample containers to avoid loss or injury due to breakage.
- 8.5 After extraction, extracts must be stored in the GC/MS refrigerator at  $4^{\circ}\text{C}$  until analysis.

## 9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

- 9.1 Graduated cylinders: 10 mL, 100 mL, 1000 mL
- 9.2 Separatory funnels with PTFE stopcocks: 2 L
- 9.3 Erlenmeyer flasks: 250 mL, 500 mL
- 9.4 Kuderna-Danish (K-D) concentrator glassware
- 9.4.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)
- 9.4.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)
- 9.4.3 Concentrator tube clips
- 9.4.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)
- 9.4.5 Snyder columns: Two-ball micro
- 9.5 pH indicator strips: pH range 0-14
- 9.6 Filter funnels: 100 mm
- 9.7 Filter funnels: 250 mL, ribbed
- 9.8 Filter paper: Fisher P8 qualitative or equivalent
- 9.9 Glass wool
- 9.10 Pasteur pipettes: Glass disposable, 2 mL
- 9.11 Water bath: Capable of variable temperature control to  $\pm 5^{\circ}\text{C}$
- 9.12 Hot plate: Capable of variable temperature control to  $\pm 5^{\circ}\text{C}$

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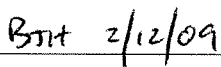
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- 9.13 Beakers, glass: 100 mL, 400 mL and 800 mL
- 9.14 Boiling chips: PTFE, rinsed with methylene chloride
- 9.15 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L
- 9.16 Vials with PTFE-lined screw-cap lids: Amber, 40 mL
- 9.17 Syringes, micro gastight: 100 uL and 1000 uL
- 9.18 Centrifuge tubes: PTFE, 40 mL
- 9.19 Centrifuge: Variable speed
- 9.20 Vials: Glass auto-sampler with PTFE-lined screw-cap lids, 1.7 mL calibrated to 1.0 mL (as outlined in SOP number GR-09-106)
- 9.21 Vials: Glass PTFE-lined screw-cap lids, 14 mL calibrated to 10 mL (refer to TriMatrix SOP GR-09-106)
- 9.22 Balance: Analytical, capable of accurate measurement to the nearest 0.0001 g
- 9.23 Sodium sulfate rinsing equipment
- 9.23.1 Buchner funnel: 20 cm
- 9.23.2 Flask: 4 L Vacuum
- 9.23.3 Drying pan: 13 x 9 inch, metal
- 9.23.4 Vacuum pump
- 9.23.5 Drying oven
- 9.23.6 Stopper: PTFE, with hole for the Buchner funnel
- 9.23.7 Clamps: supporting
- 9.23.8 Filter paper: qualitative fast, 20 cm
- 9.23.9 Squirt bottle: PTFE only
- 9.24 Phase separation paper (JT Baker 8002-15)

## 10.0 ROUTINE PREVENTIVE MAINTENANCE

- 10.1 Glass syringes must be cleaned or replaced when needles appear bent or plugged.

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## 11.0 CHEMICALS AND REAGENTS

### 11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above reporting limits.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride, to minimize interference from phthalate ester contamination.

11.1.2.1 To a 1000 mL separatory funnel, add 1000 mL of reagent water.

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

### 11.2 Methylene chloride: pesticide grade or equivalent

### 11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed

11.3.1 Sodium sulfate must be solvent-rinsed before use. Assemble the rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 Kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing the insides down with methylene chloride from a PTFE squirt bottle.


11.3.4 Pour the methylene chloride in the container over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride, if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from a PTFE squirt bottle. Maintain vacuum until the solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

Note: The oven must be vented to the outside to prevent methylene chloride fumes from entering the laboratory.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

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- 11.3.8 After cooling, return to the original container using a clean glass funnel.
- 11.3.9 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.
- 11.4 Sodium hydroxide solution, 10N: (prepared from ACS grade pellets)
- 11.4.1 Carefully add 400 g sodium hydroxide to 1 L of laboratory reagent water that has not been methylene chloride extracted.
- 11.4.2 Stir on a magnetic stir plate until dissolved then store in a closed and labeled glass container.
- Caution:** Adding sodium hydroxide to water will generate significant heat. Add pellets slowly and cool to room temperature before using.
- 11.5 Sulfuric acid solution, 1:1 (v/v): Reagent ACS
- 11.5.1 Slowly add 500 mL of concentrated sulfuric acid to 500 mL of laboratory reagent water that has not been extracted with methylene chloride.
- 11.5.2 Stir carefully when cool then store in a closed, labeled glass container.
- Caution:** Adding sulfuric acid will generate significant heat. Add slowly to avoid splattering and cool before using. **ALWAYS ADD SULFURIC ACID TO WATER (NEVER ADD WATER TO SULFURIC ACID).**
- 11.6 Methanol: pesticide grade or equivalent
- 12.0 STANDARDS PREPARATION**
- 12.1 Laboratory-made surrogates and spike solutions are made by the following rules:
- 12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.
- 12.1.2 Use the appropriate solvent for dilution.
- 12.1.3 Label all glassware and vials holding surrogate/spiking solutions.
- 12.1.4 Use minimal headspace in vials.
- 12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:
- 12.2.1 Surrogate or spike name
- 12.2.2 Laboratory-assigned surrogate or spike ID

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


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- 12.2.3 Date prepared
- 12.2.4 Analyst initials
- 12.2.5 Solvent used
- 12.2.6 Concentration and units
- 12.2.7 Expiration date
- 12.3 Surrogate/spike data are recorded in an extraction standards logbook. Solutions are labeled with a two-letter prefix followed by a number depending upon logbook page and line. Surrogate/spiking solutions are stored in the prep lab refrigerator at 4° ±2° C. A dilution of spiking solution must be analyzed by the GC/MS laboratory to check concentration before use by the extraction lab. Concentrations must be within established laboratory acceptance limits of 80-120%.
- 12.4 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If neat compound purity is below 95%, concentration must be accounted for in analytical calculations. Locate neat chemicals within the laboratory by use of the chemical inventory list. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered.
- 12.4.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Over-sonicating will generate excessive heat. Dilute to volume and mix well by inverting at least three times. Discard the volume in the neck of the flask and transfer to pre-labeled vials. Store the vials at 4° ±2° C.
- 12.4.2 For neat liquids, weigh into a tared volumetric flask half-full of appropriate solvent. Add dropwise using a Pasteur pipette directly into the solvent, being careful not to touch the sides of the flask. Record the mass, swirl then dilute to volume. Mix well and discard the volume in the neck of the flask. Transfer to pre-labeled vials and store at 4° ±2° C.
- 12.5 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however, if a manufacturer's expiration date occurs within that time for neat materials used. Laboratory-assigned expiration dates cannot exceed the manufacturer's expiration date. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.
- 12.6 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration.

$$10000 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 100 \text{ mg/L}$$

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$$100 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 1.0 \text{ mg/L}$$

- 12.7 When making a dilution, all data must be entered into Element™.
- 12.7.1 If a dilution of a dilution is made then the resultant solution would be given another number.
- 12.7.2 For solution preparation, use only pesticide-grade or better solvents.
- 12.7.3 Before using a solution, calculations performed when calculating concentration must be verified by a second analyst. Once calculations have been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a solution cannot be used. Acceptance limits are based on 80% to 120% of calculated values.
- 12.8 At the end of a shift, vials containing 5 mL or less must be disposed of to minimize contamination and concentration in the solution.
- 12.9 Acid and base compound surrogate solutions are made separately, from neat materials.
- 12.9.1 Add 0.5 g of each base-neutral surrogate compound to a 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.
- 12.9.2 Add 1.0 g of each acid surrogate compound to another 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.
- 12.9.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. Have this solution checked by GC/MS. Once approved, the prep lab and the mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.
- 12.9.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Surrogate	Mass (g)	Dilution Volume (mL)	Stock Concentration (ug/mL)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d <sub>5</sub>	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d <sub>6</sub>	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

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12.10 Spike solutions with reference to EPA Method 8270C are purchased in individual base/neutral and acid ampules except for naphthalene, which is diluted in methylene chloride from the neat compound.

12.10.1 Weigh 1.0 g of naphthalene into a 100 mL volumetric flask. Add approximately half-volume of methylene chloride to the flask and swirl, shake or sonicate to dissolve then dilute to volume with methylene chloride. Concentration of this solution is 10,000 ug/mL naphthalene.

12.10.2 Pipette 1.0 mL of acid ampule #1 into a 50 mL volumetric flask half-filled with methanol.

12.10.3 Into the same 50 mL volumetric flask, pipette 1.0 mL of base/neutral ampule #2.

12.10.4 Pipette 0.5 mL of the naphthalene solution into the flask then dilute to volume with methanol.

12.10.5 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.10.6 Transfer the entire solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Compound	Mass	Dilution Volume (mL)	Stock/Ampule Concentration (ug/mL)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Naphthalene	1.0 g	100(MeCl <sub>2</sub> )	10000	2.0:200	100
4-Chloro-3-methylphenol (#1)			7500	5.3:200	200
2-Chlorophenol (#1)			7500	5.3:200	200
4-Nitrophenol (#1)			7500	5.3:200	200
Pentachlorophenol (#1)			7500	5.3:200	200
Phenol (#1)			7500	5.3:200	200
Acenaphthene (#2)		5000	4.0:200	100	
1,4-Dichlorobenzene (#2)			5000	4.0:200	100
2,4-Dinitrotoluene (#2)			5000	4.0:200	100
N-Nitrosodi-n-propylamine (#2)			5000	4.0:200	100
Pyrene (#2)			5000	4.0:200	100
1,2,4-Trichlorobenzene (#2)			5000	4.0:200	100

12.11 Spike solutions with reference to EPA Method 625 are purchased from NSI (Catalog # C-397) at 100 ug/mL. These solutions are used as received and expiration is 6 months from date received. No dilution is required.

12.12 Spike solution with compounds for reference to SW-846 Method 1311 is purchased from Ultra Scientific (Catalog # TCLP-512).

12.12.1 Pipette 1250 µL of 2000 ug/mL purchased solution into a 25 mL volumetric flask half-filled with methanol and dilute to volume.

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12.12.2 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.12.3 Transfer the entire solution to a 40 mL amber vial with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Compound	Ampule Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (mg/L)
o-cresol	2000	1.25:25	100
m-cresol	2000	1.25:25	100
p-cresol	2000	1.25:25	100
1,4-dichlorobenzene	2000	1.25:25	100
2,4-dinitrotoluene	2000	1.25:25	100
hexachlorobenzene	2000	1.25:25	100
hexachlorobutadiene	2000	1.25:25	100
hexachloroethane	2000	1.25:25	100
nitrobenzene	2000	1.25:25	100
pentachlorophenol	2000	1.25:25	100
pyridine	2000	1.25:25	100
2,4,5-trichlorophenol	2000	1.25:25	100
2,4,6-trichlorophenol	2000	1.25:25	100

12.13 Additional spiking solutions for client-specific analytes will be prepared as needed.

### 13.0 ANALYTICAL PROCEDURE

13.1 Rinse all glassware with methylene chloride before use.

13.2 Label each Erlenmeyer flask with the following information:

13.2.1 Sample number

13.2.2 "S" for surrogate

13.2.3 "A" for acid when appropriate

13.2.4 "B" for base when appropriate

13.2.5 "L" for spike when appropriate

13.2.6 Method

13.2.7 Final volume

13.3 Label Kuderna-Danish (K-D) flasks and concentrator tubes:

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- 13.3.1 Sample number
- 13.3.2 Parameter (BNA, TCLP, as appropriate)
- 13.3.3 Final volume
- 13.4 Determine sample volume as outlined below.
- 13.4.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark then measure with a graduated cylinder to attain the sample volume.
- 13.4.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. **DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE.** After sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.
- 13.4.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting. Extracting less than 500 mL will result in elevated reporting limits, which must be client approved.
- Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.
- 13.5 Add 100  $\mu$ L of surrogate solution to each separatory funnel with a 100  $\mu$ L syringe, to extracts taken to 1.0 mL final volume. Add 1.0 mL of surrogate to each separatory funnel with a 1000  $\mu$ L syringe, to extracts taken to 10.0 mL final volume.
- 13.5.1 Syringes used must be dedicated to BNA surrogate additions.
- 13.5.2 Surrogate is added to all extractions including samples, Extraction Blanks (BLK), Blank Spikes (BS) and Matrix Spikes.
- 13.5.3 Do not submerge the syringe into a liquid when injecting surrogate. Slowly inject approximately one inch above the liquid's surface.
- 13.5.4 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.
- 13.5.5 Cross out the "S" on each corresponding Erlenmeyer flask as surrogate is added.
- 13.6 Add 100  $\mu$ L of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 1.0 mL. Add 1.0 mL of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 10.0 mL. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

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- 13.7 Acidify samples by adding approximately 2 mL of 1:1 sulfuric acid solution to each separatory funnel using a Pasteur pipette. Mix well by swirling and/or shaking.
- 13.8 Rinse the sample jar and/or graduated cylinder with methylene chloride:
- 13.8.1 Add approximately 100 mL methylene chloride to each sample jar. Replace the lid (very important).
  - 13.8.2 Shake and vent as necessary. If a graduated cylinder was used to measure the sample volume, transfer the methylene chloride from the sample container into the graduated cylinder. Swirl the methylene chloride to rinse the cylinder sides.
  - 13.8.3 Transfer the methylene chloride into the separatory funnel containing sample.
- 13.9 Acid Extraction:
- 13.9.1 Stopper the separatory funnel and invert once. Immediately open the stopcock to release solvent pressure.
  - 13.9.2 Close the stopcock, shake and vent again.
  - 13.9.3 Repeat until there is no evidence of pressure.
  - 13.9.4 Shake for one to two minutes.
  - 13.9.5 Let the separatory funnel sit for at least ten minutes for phase separation.
    - 13.9.5.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into a separatory funnel to take a reading.
    - 13.9.5.2 The pH should be less than 2. If not, add additional 2 mL aliquots of acid until a pH of less than 2 is attained then cross out the "A" on the corresponding Erlenmeyer flask. Add no more acid than one percent of the initial sample volume (10 mL for a 1000 mL initial volume) as larger additions constitute a significant extraction volume change.
    - 13.9.5.3 Consult with the project chemist if a sample cannot be adjusted to a pH less than 2 with the maximum acid volume.
  - 13.9.6 If a heavy emulsion has formed, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.
    - 13.9.6.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.

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- 13.9.6.2 If separation does not occur, drain the entire volume from the 125 mL funnel into a PTFE centrifuge tube (this may require multiple tubes). Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.
- 13.9.7 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the corresponding 500 mL Erlenmeyer flask, passing it through phase separation paper. Rinse the paper thoroughly.
- 13.9.8 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.
- 13.10 Repeat the extraction twice more, each time using approximately 60 mL of methylene chloride.
- 13.10.1 Repeat the process of rinsing the sample container and (if used) graduated cylinder with methylene chloride, then transfer to the separatory funnel for the second extraction.
- 13.10.2 Add the methylene chloride directly to the separatory funnel for the third extraction.
- 13.10.3 Approximately 200 mL of acid solvent extract will be collected.
- 13.11 After the acid extraction is complete, adjust pH to greater than 11 by adding approximately 7 mL of 10N sodium hydroxide solution to each separatory funnel using a graduated cylinder. Mix well by swirling and/or shaking.
- 13.12 Base-Neutral Extraction
- 13.12.1 Add 60 mL of methylene chloride.
- 13.12.2 Stopper the separatory funnel and invert. Immediately open the stopcock to release pressure.
- 13.12.3 Close the stopcock, shake and vent again.
- 13.12.4 Repeat until there is no evidence of pressure venting.
- 13.12.5 Shake for an additional two minutes.
- 13.12.6 Let the separatory funnel sit for at least ten minutes, for phase separation.
- 13.12.6.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into separatory funnel to take a reading.
- 13.12.6.2 The pH should be greater than 11. If not, add additional 1 mL aliquots of sodium hydroxide until a pH of greater than 11 is attained, then cross out the "B" on the corresponding Erlenmeyer flask. Add no more than one percent of the initial extraction volume (10 mL for a 1000 mL initial volume) as larger additions constitute sample dilution.

Approved By: MA 2-12-09  
QA Officer

Approved By: BSH 2/12/09  
Area Supervisor



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- 13.12.6.3 Consult with the project chemist if a sample cannot be adjusted to a pH greater than 11 with the maximum base volume.
- 13.12.7 If a heavy emulsion forms during extraction, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.
- 13.12.7.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.
- 13.12.7.2 If separation does not occur, drain the entire contents of the 125 mL funnel into a PTFE centrifuge tube. Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.
- 13.12.8 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the 500 mL Erlenmeyer flask containing acid compound extract, passing it through phase separation paper. Rinse the paper thoroughly.
- 13.12.9 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.
- 13.13 Repeat the base-neutral extraction twice more, each time using approximately 60 mL of methylene chloride. A total of approximately 400 mL of BNA extract will be collected for concentration.
- 13.14 When extractions are complete, if a graduated cylinder was not used to determine sample volume, determine the volume by filling the sample container with tap water up to the water level mark. Transfer to a graduated cylinder and record the volume.
- 13.15 Remove all sample identification tags from the empty sample jar and discard both.
- 13.16 Transfer extract to a K-D concentrator:
- 13.16.1 Quantitatively transfer each extract from the 500 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into Kuderna-Danish concentrators, with an attached 10 mL graduated concentrator tube.
- 13.16.2 Rinse the flask and funnel with several 20-30 mL aliquots of methylene chloride to complete the transfer.
- 13.17 Concentrate the extracts
- 13.17.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.
- 13.17.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

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- 13.17.3 Place the concentrators in a water bath (70-80° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.
- 13.17.4 At the proper rate of distillation, the column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder columns periodically.
- 13.17.5 When the apparent volume of liquid reaches 3-4 mL (in ten to fifteen minutes), remove the K-D and let drain for at least 10 minutes.
- 13.17.6 Remove the Snyder column, rinsing the lower joint into the concentrator tube with 2 to 3 mL of methylene chloride.
- 13.18 Micro-Snyder column technique is used to adjust final extract volumes to 1.0 mL. Blowdown is not used when adjusting final volume to 1.0 mL, to minimize analyte loss from concentration.
- 13.18.1 Micro-Snyder Column Technique:
- 13.18.1.1 Add another clean boiling chip then attach a 2-ball micro-Snyder column.
- 13.18.1.2 Pre-wet the column with 0.5 mL methylene chloride.
- 13.18.1.3 Place concentrator tubes in a water bath, partially immersed. Water temperature should be 60-70° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.
- 13.18.1.4 When the apparent volume reaches 0.5 mL, remove from the bath. Cool for at least ten minutes.
- 13.18.1.5 Rinse the ground-glass joints with 0.2 mL methylene chloride after removing Snyder columns.
- 13.18.1.6 Transfer extracts to a 1.0 mL calibrated vial using a clean disposable Pasteur pipette.
- 13.18.1.7 Using approximately 0.2 mL of methylene chloride, rinse each concentrator tube, then transfer to the vial.
- 13.18.1.8 Adjust the final volume to 1.0 mL with methylene chloride then cap tightly.
- 13.18.1.9 Extracts are ready for analysis and must be stored in the GC/MS sample refrigerator at 4 ±2° C.

#### 14.0 DATA REPORTING AND DELIVERABLES

- 14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

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QA Officer

Approved By: BT 2/12/09  
Area Supervisor



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14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

## 15.0 QUALITY ASSURANCE

15.1 Extraction analysts must extract an Extraction Blank and Blank Spike daily, once per shift or every 20 samples (whichever is more frequent) to demonstrate that extraction interferences are under control.

15.2 Matrix Spikes and Matrix Spike Duplicates (provided enough sample volume is received) are extracted at a minimum of once every twenty samples or once a week, depending upon the frequency of sample receipt.

15.2.1 Performance records are maintained to document data quality generated.

15.2.2 Matrix spiked samples are prepared for analysis by first adding spike solution and surrogate before extraction.

15.3 Blank Spikes are prepared by extracting the addition of BNA spike solution and surrogate solution to 1 L of pre-extracted laboratory reagent water.

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation and re-extraction may be necessary if surrogate or spiked compounds fail to pass laboratory established acceptance limits.

15.5 Surrogate must be added to all extractions.

## 16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting a demonstration of capability study.

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- 16.2 Prepare a spiking solution that will give final extract concentrations, mid-range to the GC/MS calibration. This solution must be prepared independent of GC/MS calibration standards. Prepare four (4) 1000 mL aliquots of pre-extracted water. Inject 100  $\mu$ L of spiking solution into each. Add surrogate, then extract as samples, going through all extraction steps in this procedure, to a final volume of 1.0 mL.
- 16.3 After extraction, have the four (4) extracts analyzed for all spiked compounds in accordance with all GC/MS procedure requirements.
- 16.4 Input results to the IDC spreadsheet to calculate average percent recovery (x) and percent relative standard deviation of recovery (s), for each analyte, using the four results.
- 16.4.1 For each analyte, (x) must be within historical statistical recovery limits and (s) must be less than or equal to 20%. If (s) and (x) for all analytes meet acceptance, the demonstration of capability study is acceptable. The analyst is authorized to extract samples by the procedure.
- 16.4.2 If one or more analytes fail at least one acceptance limit, the analyst must proceed through the following steps.
- 16.4.2.1 Locate and correct any source of error then repeat the demonstration of capability only for analytes not meeting acceptance. If the failed analytes are acceptable on the second attempt, the study is complete and acceptable.
- 16.4.2.2 Repeated failure will indicate general problems with the extraction procedure or analyst technique. If this occurs, correct and revise any faulty steps in the procedure or technique.
- 16.4.2.3 Once corrective action has been taken, re-extract the demonstration of capability study for all analytes. If all analytes are acceptable, the study is complete and acceptable.
- 16.5 Samples may not be prepared by any analyst until an acceptable demonstration of capability study has been successfully completed.
- 16.6 A successful continuing demonstration of capability (CDC) study must be completed annually by all analysts by one of the following approaches:
- 16.6.1 By repeating the IDC study.
- 16.6.2 By using four consecutive blank spike results obtained during the course of routine sample extraction and when extracted exclusively by the analyst.
- 16.6.3 By extracting an acceptable blind performance testing sample.
- 16.6.4 By exclusively extracting a method detection limit (MDL) study. The last four results of the study may be used.
- 16.7 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

Approved By: MB 2-12-09  
QA Officer

Approved By: BST 2/12/09  
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## 17.0 POLLUTION PREVENTION

- 17.1 Maintain an inventory of all chemicals used in the laboratory to monitor chemical use.
- 17.2 Reference any specific written disposal instructions before disposing of laboratory chemicals.
- 17.3 Conserve the use of chemicals where applicable
- 17.4 Comply with all environmental laws associated with chemicals in the laboratory.

## 18.0 WASTE MANAGEMENT

- 18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.
- 18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.
- 18.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 for laboratory waste disposal requirements.

## 19.0 REFERENCES

- 19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition*. Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"
- 19.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

## 20.0 ATTACHMENTS

- 20.1 Preparation Batch Report Example
- 20.2 Standards Log Report Example
- 20.3 Water Extraction Logbook Example

Approved By:           m 2-12-09            
QA Officer

Approved By:           BTt 2/12/09            
Area Supervisor





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**Attachment 20.1**  
**Preparation Batch Report Example**

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0801114** Page 1 of 1

Printed: 11-7-2008 10:14:54AM

Semivolatiles MS, Waste Water, 625 Liquid/Liquid Extraction

Batch comments:

(none)

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Spike ID	uL Spike	Comments
0801114-BLK1		Feb-01-08 08:23	KB9	1000	5				
0801114-BLK2		Feb-05-08 08:23	ASC	1000	1				same as batch 0801235
0801114-BS1		Feb-01-08 08:23	KB9	1000	5		8010850	500	
0801114-BS2		Feb-05-08 08:23	ASC	1000	1		8010850	100	same as batch 0801235
0801114-BSD1		Feb-01-08 08:23	KB9	1000	5		8010850	500	
0801114-DUP1		Feb-01-08 08:23	KB9	1000	5	0801501-19			
0801114-DUP2		Feb-01-08 08:23	KB9	1000	5	0801501-20			
0801114-DUP3		Feb-01-08 08:23	KB9	1000	5	0801501-25			
0801114-DUP4		Feb-01-08 08:23	KB9	1000	5	0801501-26			
0801114-DUP5		Feb-05-08 08:23	ASC	1000	1	0801501-27			
0801501-19	A	Feb-01-08 08:23	KB9	1000	5				Added for BatchQC in 0801114 BatchQC
625 SPOCs (master list) 625 SPOCs (TTOs) 625 SPOCs (TTOs) custom									
0801501-20	A	Feb-01-08 08:23	KB9	1000	5				Added for BatchQC in 0801114 BatchQC
625 SPOCs (master list) 625 SPOCs (TTOs) 625 SPOCs (TTOs) custom									
0801501-25	A	Feb-01-08 08:23	KB9	1000	5				acids, base neutrals acids, base neutrals
625 SPOCs (master list) 625 SPOCs (TTOs) 625 SPOCs (TTOs) custom									
0801501-26	A	Feb-01-08 08:23	KB9	1000	5				acids, base neutrals acids, base neutrals
625 SPOCs (master list) 625 SPOCs (TTOs) 625 SPOCs (TTOs) custom									
0801501-27	A	Feb-05-08 08:23	ASC	1000	1				Added for BatchQC in 0801114 BatchQC
625 SPOCs (master list) 625 SPOCs (TTOs) 625 SPOCs (TTOs) custom									

Comments	Analyst Initials
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bch\_TM\_byAnalysis.rpt

Approved By: 20 2-12-09  
QA Officer

Approved By: Brt 2/12/09  
Area Supervisor



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**Attachment 20.2**  
**Standards Log Report Example**

**Analytical Standard Record**

**TriMatrix Laboratories, Inc.**

**8060471**

Description	BNA Surrogate	Expires	Nov-10-08
Standard Type	Surrogate Spike	Prepared	Jun-16-08
Solvent	MeOH Lot#076070	Prepared By	Andrea S. Colborn
Final Volume (mls)	2000	Department	Semivolatiles MS
Vials	1	Last Edit	Jun-16-08 17:19 by ASC

Analyte	CAS Number	Concentration	Units
Phenol-d6	13127-88-3	200	ug/mL
o-Terphenyl	84-15-1	100	ug/mL
Nitrobenzene-d5	4165-60-0	104	ug/mL
Decafluorobiphenyl	434-90-2	100	ug/mL
2-Fluorophenol	367-12-4	202	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	201	ug/mL

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7120351	Base Surrogate Stock	Dec-14-07	Brian J. Hall	Dec-14-08	Dec-14-07 11:52 by ASC	100
7120371	Acid Surrogate Stock	Dec-18-07	Andrea S. Colborn	Nov-10-08	Dec-18-07 08:40 by ASC	100

Approved By: MS 2-12-09  
 QA Officer

Approved By: Bret 2/12/09  
 Area Supervisor



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**Attachment 20.3**  
**Water Extraction Logbook Example**



**Organic Extraction Lab**  
**Water Extraction Logbook**

Date: 12/29/03

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume	Matrix Spike Number and Volume
RMT	36416-13	352226-233	211038	82708N 3510	PL4.2-7	PL3.12-7
Rose Top	29928-597	352327-328	"	82708N 3510	"	"
RMT	36416-13	352228-270	210615	8015 DEC 3510		GL 2.41-16
N/A 12/29/03						

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
352226	82	990	1.0mL	MBS					
352227	↓	1040	↓	↓					
352228	↓	1040	↓	↓					
352231	↓	1060	↓	↓					
352233	↓	1060	↓	↓					
MPB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
352327	82	1030	1.0mL	MBS					
352328	↓	1040	↓	↓					
352228	82	1020	1.0mL	MBS					
352230	↓	255	↓	↓					
MPB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
N/A 12/29/03									

Reagent Information	
CH <sub>2</sub> Cl <sub>2</sub> Lot #: 030986	1:1 H <sub>2</sub> SO <sub>4</sub> Reagent #: PLR1.42-13
Hexane Lot #: 030702	1:1 HCl Reagent #: NA
Na <sub>2</sub> SO <sub>4</sub> Reagent #: PLR1.42-16	10N NaOH Reagent #: PLR1.44-11
	NaCl Reagent #: NA

file: Prep Lab Water Logbook

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revision: 0.0

Approved By: MD 2-12-09  
 QA Officer

Approved By: BST 2/12/09  
 Area Supervisor